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INVESTIGATION OF THE RELATIONSHIP OF TWINNING TO BRITTLE FRACTURE OF REFRACTORY METALS

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April 1963

Directorate of Materials and Processes
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NOTICES

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FOREWORD

This report was prepared by the Battelle Memorial Institute, Columbus, Ohio under USAF Contract No. AF 33(657)-7376. This contract was initiated under Project No. 7351, "Metallic Materials", Task No. 735101, "Refractory Metals". The work was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division. Mr. C. S. Hartley was the project engineer.

This report covers work from November 1, 1961, to December 31, 1962.

ABSTRACT

The first year's work on this program is reported. It is concluded that there is good evidence for brittle fracture induced by mechanical twinning in single crystals and polycrystalline Mo-C with grain sizes larger than 0.40 mm. However there is no evidence for such failures in Mo-C of grain size smaller than 0.25 mm, or in high-purity Cb. The effect of recrystallization texture on twinning is demonstrated, and the orientation dependence of twinning in Cb crystals is reported; a most unusual plastic instability is described in crystals of one orientation. It is shown that twinning in Mo-35Re occurs in bursts, which form in less than 2 μsec; nucleation of twins occurs only at small strains but lateral growth takes place up to at least 12 per cent strain.

This technical documentary report has been reviewed and is approved.

I. PERLMUTTER
Chief, Physical Metallurgy Branch
Metals and Ceramics Laboratory
Directorate of Materials and Processes

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INVESTIGATION OF THE RELATIONSHIP OF TWINNING TO BRITTLE FRACTURE OF REFRACTORY METALS

by

C. N. Reid, A. Gilbert, and G. T. Hahn

INTRODUCTION

There is great contemporary interest in the refractory metals and alloys as advanced structural materials. Good evidence exists that mechanical twinning is capable of creating inter- and transcrystalline cracks in bcc metals, and this must be considered as a possible cause of brittleness in the refractory metals. This research program is intended to produce an appraisal of the evidence for twin-induced fracture among refractory metals, and an understanding of the factors which control its occurrence.

REVIEW OF THE LITERATURE

Since twinning provides the means for this type of failure, it is desirable first to review current knowledge of the twinning phenomenon itself.

Mechanical twins are produced in body-centered cubic metals by deformation at low temperatures, or at high loading rates. Twins have been observed in iron[1] and its binary alloys with silicon[2], phosphorus[3], molybdenum[3], and aluminum[4]; in the Group V-A metals vanadium[5], columbium[6], and tantalum[7], and in the Group VI-A elements chromium[8], molybdenum[9], and tungsten[10], and their binary alloys with rhenium[11,12].

Twinning Crystallography

The twins consist of regions of crystal in which a particular set of $\{112\}$ planes is homogeneously sheared by 0.707 in a $<111>$ direction. The same atomic arrangement may be visualized by a shear of 1.414 in the reverse $<111>$ direction, but this larger shear has never been observed. As a consequence of this, twinning systems, as specified by a plane and a direction, are "polarized", the material shears only in one sense of this direction. There are twelve different twinning systems, and these are labeled and illustrated in Figure 1 according to the convention of Schmid and Boas[13]. Their "polarization" implies that for a given orientation of the crystal relative to a uniaxial stress only x systems may operate under tensile loading, and only the remaining (12-x) systems under compression. Figure 2 taken from Allen et al[14] illustrates the twinning systems that are operative in a crystal under tension or compression as a function of the direction of the applied uniaxial stress.

$\bar{1}00$

$\bar{2}\bar{1}1$
●
9

$\bar{2}11$
●
3

$\bar{1}\bar{2}1$
●
11

△
$\bar{1}\bar{1}1$

△
$\bar{1}11$

$\bar{1}21$
●
5

$\bar{1}\bar{1}2$
●
7

$\bar{1}12$
●
1

o$\bar{1}$o □

□ $\bar{0}01$

□ 010

8
●
1$\bar{1}$2

2
●
11$\bar{2}$

12
●
1$\bar{2}$1

6
●
121

△
1$\bar{1}$1

△
111

10
●
2$\bar{1}$1

4
●
211

100
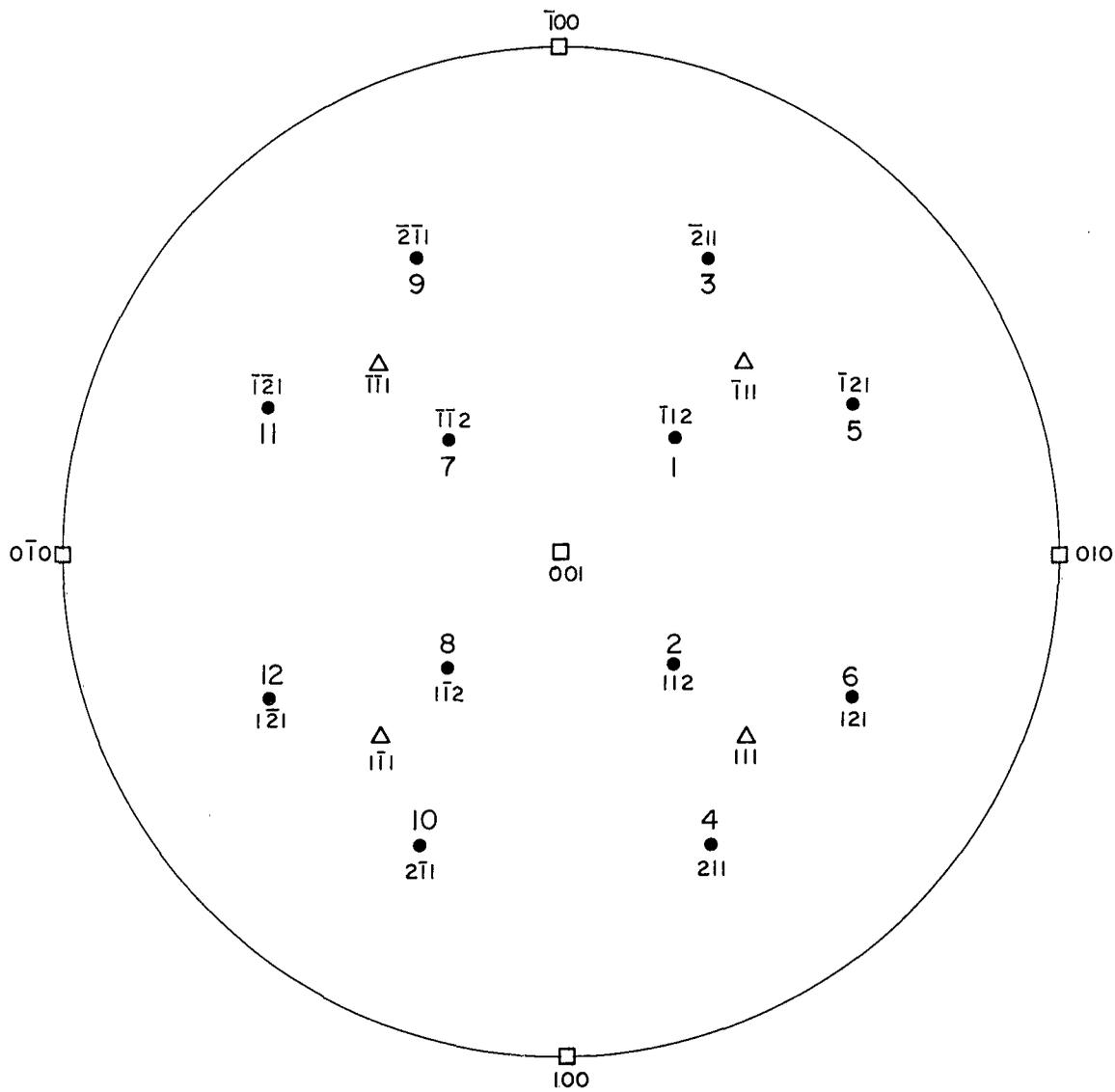
FIGURE 1. TWINNING SYSTEMS IN BCC CRYSTALS NUMBERED ACCORDING TO THE CONVENTION OF SCHMID AND BOAS

B [Ī11]

F [Ī12]

D [Ī13]

A [00Ī]   E [012]   C [01Ī]
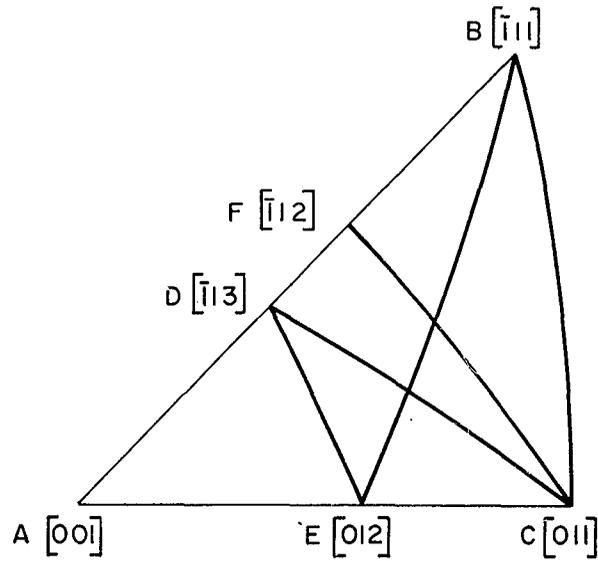
A-44184

Stressed in tension systems $\left\{\begin{array}{c} 2,7,8 \\ 6,9 \\ 1 \\ 10 \\ 12 \\ 11 \end{array}\right\}$ operate when the orientation is within $\left\{\begin{array}{l} ABC \\ BCF \\ ACF \\ BCD \\ BDEC \\ BEC \end{array}\right.$

Stressed in compression systems $\left\{\begin{array}{c} 3,4,5 \\ 1 \\ 11 \\ 6,9 \\ 10 \\ 12 \end{array}\right\}$ operate when the orentiation is within $\left\{\begin{array}{l} ABC \\ BCF \\ BEA \\ ACF \\ ADC \\ ADE \end{array}\right.$

FIGURE 2.   THE OPERATIVE TWINNING SYSTEMS AS A FUNCTION
OF CRYSTAL ORIENTATION[14]

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The Strain Contribution of Twinning

The ultimate extent to which twinning may contribute to the strain of a specimen occurs in the case where a crystal of optimum orientation is totally transformed into a twin. According to Schmid and Boas[13] the maximum longitudinal strain ϵ_{max} and the optimum orientation are given by maximizing the equation:

$$\epsilon_{max} = \sqrt{1 + 2s \sin \chi \cos \lambda + s^2 \sin^2 \chi} \quad , \tag{1}$$

where s is the shear involved in twinning, and χ and λ are the angles between the longitudinal specimen axis and the twinning plane and direction, respectively. ϵ_{max} has the value of 41.4 per cent, when $\chi = \lambda = 54.7$ degrees. In general, however, only a fraction of the matrix transforms into a twin. Geil and Carwile[15] made some qualitative observations of twinning in polycrystalline α-Fe; they reported that twins were initiated only in the early stages of deformation, and that their thickness subsequently increased with further tensile strain until fracture of the specimen occurred.

The Morphology of Twins

The shapes of twins may be revealed in two dimensions by etching. Both the coherent and noncoherent segments of a mechanical twin boundary etch as readily as grain boundaries, and it is concluded that the twin-boundary energy is high. This has been confirmed by transmission electron microscopy which has revealed a high concentration of dislocations at boundaries[16]. Usually a twinned region is extensive in the twinning plane, being limited by the dimensions of the crystal and obstacles such as other twins, but perpendicular to this plane the region is narrow. The twin-matrix interface may have various shapes; it may be smooth with a gradual taper towards the edges of the twinned layer, having the shape of a bi-convex or plane-convex lens. Alternatively the interface may have a complicated fine structure of serrations, undulations, or flamelike features. For example, the sectioning of tungsten[17], molybdenum[18], and columbium[6] parallel and perpendicular to the twinning direction has revealed that the twins present consist of sets of parallel prisms whose long dimension is parallel to the twinning direction.

Apart from this variety of boundaries observed in the interiors of bcc metals, Hull[2] and Sleeswyk[19] have reported observations of eruptions on free surfaces of iron-silicon and iron after impingement by growing twins. They observed that in iron and iron-silicon the twin boundary making an acute angle with the surface gave rise to zig-zag indentations of the surface, and they offered explanations in terms of accommodation slip. However it is emphasized that these serrations are not twin-matrix interfaces but a pattern of slip lines.

The Shear Stress for Twinning

For a long time it has been anticipated that there is a critical shear stress responsible for twinning. This is predicted by both the dislocation theory, and the theory of homogeneous twin formation. However experimental studies of the critical stress for twinning in macroscopic crystals of iron[14] and zinc[20] contradict this; the critical stress varies with crystal orientation[14] and with specimen size[20]. Similarly, a

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variable twinning stress has been observed in zinc whiskers[21], but the average stress level was about ten times higher than that in bulk material. It is generally argued that there is a critical shear stress but that it never coincides with the nominal stress due to the presence of uncontrolled local stress concentrations within the crystals. These are believed to be more severe in bulk material than in whiskers. The work of Bell and Cahn[20] and Siems and Haasen[22] on zinc and that of Marcinkowski and Lipsitt[8] on chromium indicate that deformation by slip may be a necessary precedent of twinning. This suggests that the local stress concentrations are derived from slip dislocations.

The dislocation pile-up or queue has frequently been visualized as the source of the stress concentration. This conclusion is supported by the observation that transmission electron micrographs taken under conditions where twinning occurs show the dislocations present to be unusually straight and parallel, with a reduced number of jogs, cusps, and trails. This is the case in impacted iron[23], iron deformed slowly at 78 K[23], and Mo-35Re deformed slowly at 300 K[24]. The appearance suggests that cross slip is inhibited under these conditions; this is expected to be conducive to pile-up formation, because cross slip provides a means of escape of dislocations from developing queues.

While the validity of a critical shear stress remains to be demonstrated it is known that for a given polycrystalline material there exists a critical nominal stress for twinning. For example, it has been found that in carbon steel the stress for initial twinning is almost independent of temperature and strain rate[25]. Studies of iron-silicon[26] and mild steel[27,28] indicate that this stress depends sharply on grain size in the manner of Figure 3, but in columbium there was found to be no grain-size dependence[29] at 20 K.

The effect of interstitial impurities on the occurrence of twinning in bcc metals is disputed. The larger school[5,30-32] of thought maintains that increased purity favors deformation by twinning under given testing conditions, whereas the opposite effect has also been reported[33]. It is important to establish whether the effect of interstitials is the same when in or out of solution. The work of Edmondson[34] suggests it is not; iron crystals quenched to retain nitrogen in solution deformed by slip at 78 K but when quenched and aged causing precipitation, they deformed by twinning. Hence, it appears that interstitials in solution may inhibit twinning. While evidence is contradictory on the role of interstitial impurity, it is clear that the presence of certain substitutional elements can promote twinning most strikingly. For example, phosphorus or molybdenum in iron[3] and rhenium in chromium, molybdenum or tungsten[12] make twinning the preferred deformation mode under conditions where it did not occur before alloying.

The Kinetics of Twin Growth

From the existence of "twinning cry" it has long been believed that twins form very rapidly in bulk material. This has been confirmed in zinc by Jillson[35] and Siems and Haasen[22] using high-speed cimematography. This method primarily follows the growth in width of existing twin nuclei.

Faster techniques indicate that these nuclei form in times of the order of microseconds. For example, Förster and Scheil[26] recorded the changes in electrical

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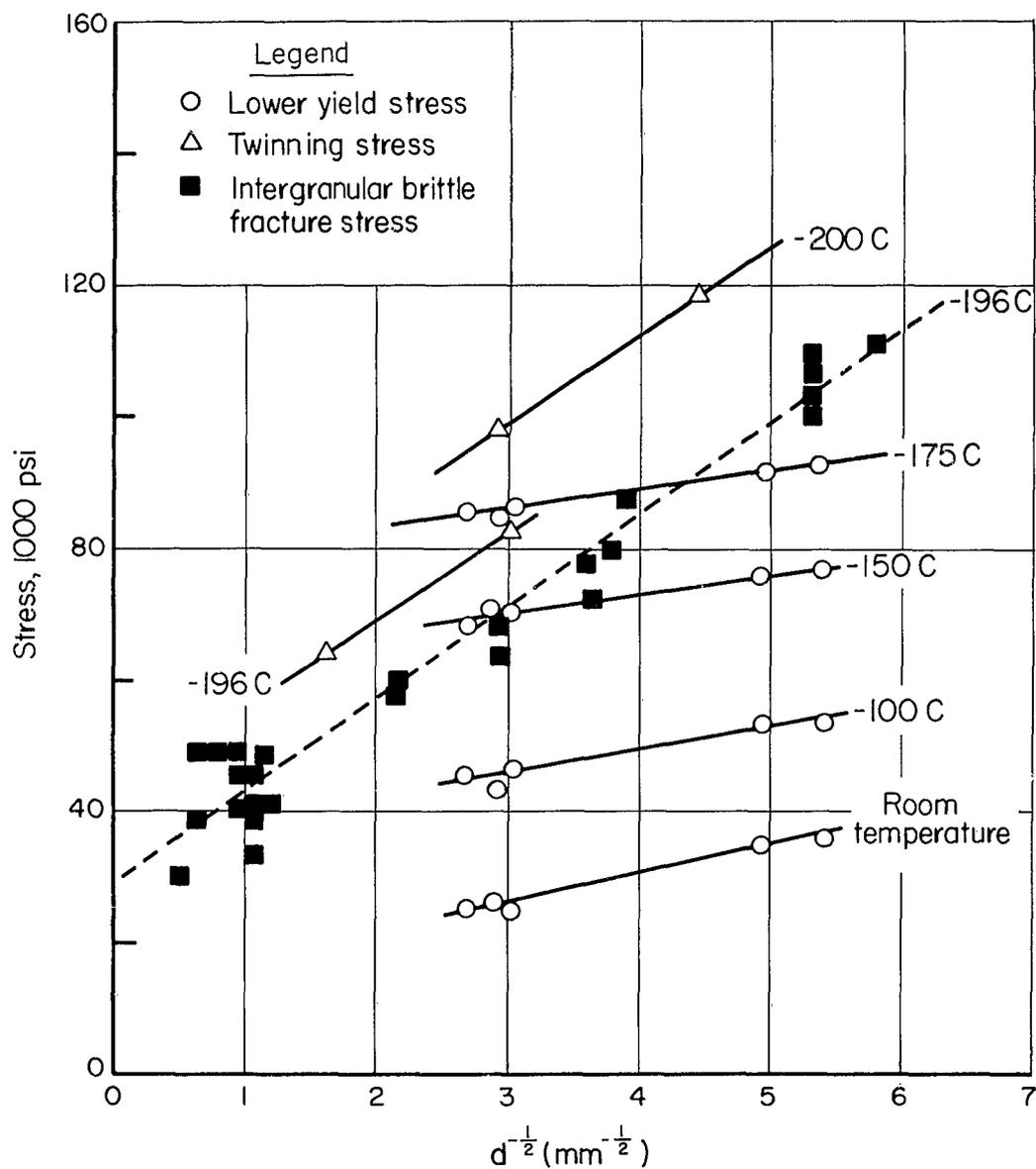
FIGURE 3. A COMPARISON OF THE GRAIN-SIZE DEPENDENCE OF TWINNING
AND LOWER YIELD STRESSES FOR IRON, ACCORDING TO
HAHN ET AL.[27] AND THE INTERGRANULAR FRACTURE STRESS,
ACCORDING TO LOW[28]

resistance which accompany twin formation in bismuth, and later Bunshah and Mehl[37] performed a similar study on bismuth, zinc, and iron. These workers identified the duration of the resistance transient with the time of formation of a twin. The transient consisted of a simple rounded step; the former authors measured durations between 5×10^{-6} and 350×10^{-6} seconds in bismuth, whereas Bunshah and Mehl measured times between 0.2×10^{-6} and 1×10^{-6} seconds in bismuth, zinc, and iron. There is a lack of agreement here in the values for bismuth.

Mason, McSkimin, and Shockley[38] followed twinning in tin by detecting changes in dimensions with a piezoelectric crystal, and Thompson and Millard[39] studied cadmium in the same way. The voltage signals from the crystals were more complicated than those from the resistance method, and therefore harder to interpret. However, the wave trains from cadmium were on the average about 100X longer than those from tin.

Recently, there have been observations by transmission electron microscopy of the formation of mechanical twins in ultrathin specimens of tin and zinc. The work of Price[21] on zinc whiskers demonstrates that twins nucleate in the absence of dislocations at sites of stress concentration at applied stresses close to the estimated theoretical twinning stress. The twins propagated at lower stresses by the nucleation and movement of twinning dislocations at slow and uniform speeds — quite unlike the cataclysm in bulk material. Fourie, et al.,[40] observed the same phenomena in thin foils of tin. However, at present it is not clear that these phenomena are also operative in bulk material.

Proposed Mechanisms of Twin Formation

There have been two fundamentally different approaches to twin nucleation. The first envisages the homogeneous formation of a thin lamellar twin of minimum stable size, in a region of locally high stress. The problem has been treated by Orowan[41] who derived an activation energy for nucleus formation which is proportional to: (shear stress)$^{-2}$. The theoretical twinning stress may be estimated as the stress which corresponds to an activation energy in the region of 1 ev. The value appropriate to zinc agrees well with that measured experimentally[21], and this may be regarded as support for the mechanism and its treatment by Orowan. The maximum conceivable rate of growth by this mechanism would be realized if the expanding loops of partial dislocation moved out across the radius of the twin lamella in quick succession at sonic velocity. For example, the time taken by a twin lamella of diameter 0.2 mm and thickness 3×10^{-4} mm in Mo-35Re is calculated to be > 30 mμsec. Thus the mechanism is consistent with very rapid twin formation.

The second approach visualizes twin formation by a heterogeneity such as a particular dislocation arrangement. Cottrell and Bilby[42] have proposed a mechanism for the bcc lattice in which a dislocation dissociates under stress to produce a mobile twinning dislocation and a sessile pole dislocation, according to the equation:

$$\frac{a}{2}[111] \rightarrow \frac{a}{3}[112] + \frac{a}{6}[11\bar{1}] \qquad (2)$$
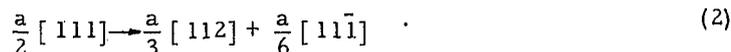
The former moves under stress about the pole along a spiral path, leaving behind a region of twinned crystal. The maximum conceivable rate of growth by this mechanism will be achieved when the fastest segments of the spiralling partial dislocation are

moving with the speed of sound.[42] If this is so, then the time taken for a twin lamella of 0.2-mm diameter and 3×10^{-4} mm thick to form in Mo-35Re would be >25 mμsec; the minimum formation time is about the same for both models. This model is able to account for the crystallography of twins observed in bcc metals, and it correctly predicts that twin growth is very rapid, once the initial dissociation is accomplished. Also, the observed slip preceding twinning may be interpreted as the generation of suitable source dislocations. However, it does not account for the other properties of twinning that have been summarized above, and there has been no direct observation of this dislocation arrangement. No clear choice between these twinning mechanisms can be made at this time.

Sleeswyk[43] has proposed a mechanism for the growth of twins along the composition plane. From energy considerations he postulates that a twin is preceded by "emissary" slip dislocations on one in every three $\{112\}$ planes, and that the remaining noncoherent twin boundary has a low energy due to the absence of a long-range stress field. The emissary dislocations and the low-energy boundary are a consequence of the dissociation of a partial dislocation:

$$-\frac{1}{6}<111> \rightarrow \frac{1}{3}<111> - \frac{1}{2}<111> \quad , \tag{3}$$

on one in every three planes parallel to the composition plane. The theory is very successful at accounting for the uniform shear that extends far beyond the twin-matrix interface, creating surface eruptions and kinks in markers such as subboundaries. Alternatively, however, it seems possible that the same effects might arise when slip dislocations in the surrounding matrix are moved by the long-range stress field of a twin.

The Relationship Between Twinning and Fracture

While there are good examples that brittle fracture of bcc metals may occur in the complete absence of twinning[44], and that twinning may occur without brittle failure[12], there have also been demonstrations that twins can produce cracks. Clearly, all three phenomena are well documented, and differences in behavior arise from variations in the material and in the experimental conditions.

Cracking at Twin Intersections

The investigations of iron-silicon by Hull[2] and Honda[45] have demonstrated that cracks are frequently created at the line of intersection of two twin lamellae in single crystals stressed in tension along a <100> direction. Edmondson[34] has observed similar behavior in iron crystals. Apparently, in order to nucleate cracks, it is necessary that the line of intersection lies in the $\{100\}$ plane situated approximately perpendicular to the applied stress. Similarly, Cahn[9] observed cracks at the intersection of twins in crystals of molybdenum deformed in compression. Sleeswyk[46] has published a model for crack formation at twin intersections which depends on an interaction of the emissary dislocations from the two twins, producing crack dislocations according to the equation:

$$\frac{1}{2}[111] + \frac{1}{2}[1\bar{1}\bar{1}] \rightarrow [100] \tag{4}$$

However, as Hull[47] has pointed out, the intersection of two simultaneously advancing twins would appear to be much less likely than the intersection of an existing twin by a growing one. Hornbogen[3] had advanced another hypothesis. He argues that since twins form very rapidly they emit stress waves which may produce cleavage cracks when reflected by an obstacle such as an intersected twin. Hornbogen has observed cracking at a twin intersection, the geometry of which is consistent with this idea.

Grain-Boundary Cracking

Studies of polycrystalline bcc metals have demonstrated that a grain boundary may part when a twin impinges upon it. In particular, Parker and Mueller[18] have published examples of this in molybdenum, and Marcinkowski and Lipsitt[8] have observed similar cases in chromium. Indirect evidence for this appears in Figure 3 which illustrates the close similarity between the twinning stress of iron and the intergranular fracture stress for mild steel. Materials containing appreciable grain-boundary segregation are perhaps the most likely candidates for this type of failure.

Microcracks at Single Twins

Small cracks have frequently been observed within twin lamellae[8] and at non-coherent irregularities on the twin-matrix interface[48]. By providing crack nuclei under conditions where propagation is favorable, these may sometimes lead to total failure, as suggested by Low[48]. Furthermore, there have been indications that a twin boundary is a preferred region for crack propagation.[10, 49]

It appears that all these cases of crack nucleation occur at sites of local stress concentration, and it is plausible to conclude that the cracks are a consequence of this magnified stress.

There have been many observations near cleavage fractures of twins whose relationship to the crack was not in any of the above categories.[33] It is strongly suspected that these twins were generated by the stress field of the propagating crack, and Bilby and Bullough[50] have justified this by calculation. In this case the crack generates the twins, the reverse of the situation that was considered above. As a rule, twins generated by the fracture are narrow, are situated in the immediate vicinity of the fracture, and are not continuous across the failure.[51] It is important in fractography to identify whether a twin is related to the fracture, and if so, whether it is a cause or an effect.

In summary, it may be said that current knowledge of mechanical twinning is very incomplete. No mechanism for nucleation and growth of twins has been fully verified, and it is not clear why the growth kinetics should differ so widely between the bulk material and electron-transparent foil. The observed relationship between the nominal twinning stress, temperature, strain rate, and grain size is not adequately understood. Furthermore the effect on twinning in bcc metals of dissolved and undissolved interstitial elements has been incompletely investigated or rationalized. Finally the concentrations of stress that are associated with twins and their intersections have been only qualitatively revealed. Clarification of any of these questions should contribute to the understanding of twin-induced fracture.

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OBJECTIVES

Molybdenum and columbium are the subjects of this investigation; they represent the extremes of ductility exhibited by the refractory metals. The primary objective is to discover the conditions under which these materials undergo twin-induced cracking. Those under study are, first, the testing conditions such as temperature and loading rate and, second, the conditions of the material such as its grain size and interstitial content. This involves a program of mechanical testing followed by metallographic examination.

Stress concentrations are associated with twin lamellae and they may be relaxed by the processes of slip, further twinning, or the generation of cracks. In bulk material, the initial growth of a twin is very rapid and the surrounding material is subjected to a high loading rate. Under these conditions, slip is limited by the mobility of the dislocations and hence twinning or cracking is favored. One of the most dangerous aspects of twinning then appears to be its kinetics of growth, and a further objective of this program has been to observe quantitatively the formation and growth of twins in a bcc material. The questions to which answers are sought include:

(1) Do twins form singly or in bursts?

(2) What is the growth rate of a twin or burst of twins?

(3) How does the contribution of twinning vary with the extent of deformation?

It was decided that this phase of the program would be conducted with the alloy Mo-35Re, enabling difficult experiments to be carried out at room temperature, and eliminating the inconvenience of a cold chamber.

EXPERIMENTAL PROGRAM

Mechanical Tests

Compression- and tension-test specimens, illustrated in Figure 4, were prepared by grinding and electropolishing from material in each condition under study. These were tested in an Instron machine, Model TT-C-L, at a given strain rate and at temperatures between 78 K and 298 K. These temperatures were attained by means of a liquid-nitrogen evaporator of modified Wessel design[52]. In addition, some tensile tests were made at 20 K in a hydraulic machine, but facilities for compression at this temperature were not available. Since there is greater ductility in compression, it was argued that comparison of behavior in tension and compression should establish whether the brittle-fracture stress can be identified with the stress for yielding or twinning. Furthermore, it was considered that compression loading would be favorable for studying crack nucleation in the absence of propagation.
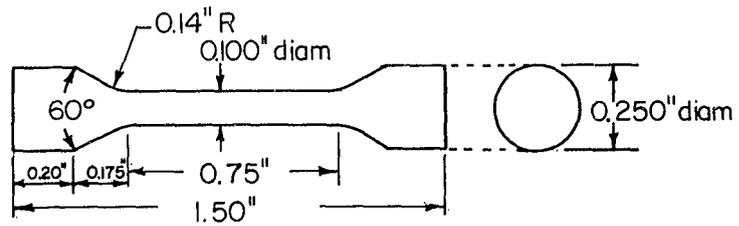
a. Round Tensile Specimen
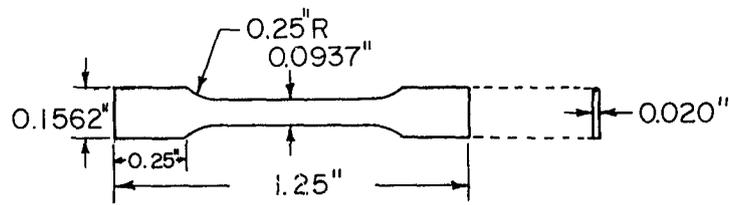
b. Sheet Tensile Specimen
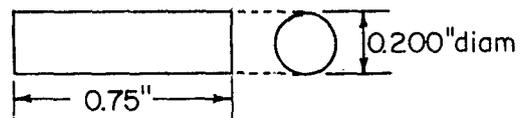
c. Compression bar   Scale - Twice actual

FIGURE 4. THE TYPES OF TEST SPECIMENS USED IN THIS INVESTIGATION

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Results for Molybdenum

Commercial arc-cast molybdenum has been investigated over a wide range of re-crystallized grain sizes. The purity level and conditions of heat treatment are summarized in Table 1. It has been estimated[53] that the dissolved interstitial content remaining in molybdenum after moderate cooling rates is as follows:

Hydrogen	Carbon	Nitrogen	Oxygen
0.1 ppm	0.1-1 ppm	1 ppm	1 ppm

It is concluded that this material was saturated with nitrogen and oxygen, and highly saturated with carbon. The grain-boundary phase that was present was therefore probably a carbide.

TABLE 1. HEAT TREATMENT OF EXPERIMENTAL MATERIALS

Material	Supplier	Supplier's Analysis, ppm	Time at Temperature, hours	Temperature, C	Cooling Rate	Atmosphere	Mean Grain Diameter, mm
Molybdenum	Universal Cyclops Steel Corporation	C: 330 N: 30 O: 15 H: 10	1/2 1/2 1	1330 1910 2000	Cool to ambient temperature in approximately 1 hour	Hydrogen Hydrogen Vacuum, 4×10^{-5} mm	0.037 0.25 0.40
Columbium	Wah Chang Corporation	C: 30 N: 56 O: 60 H: 3	1	1500	Ditto	Vacuum, 1×10^{-5} mm	0.38
Mo-35 at.% Re (first order)	Chase Brass and Copper Company		1	1800	"	Vacuum, $<5 \times 10^{-5}$ mm	0.20
Mo-35 at.% Re (later order)	Chase Brass and Copper Company		1	1800	"	Vacuum, $<5 \times 10^{-5}$ mm	0.11

Mechanical tests on molybdenum were conducted at a strain rate of 2.7×10^{-2} per min. All specimens tested in tension at 173 K and below underwent a preyield brittle fracture.

Tests on Molybdenum of Mean Grain Diameter d = 0.037 Mm. Grain sizes were measured by lineal counting of grain boundary intercepts on polished and etched sections of the specimens. The stress parameters measured when molybdenum with the smallest grain size was tested are given in Figure 5. Similar stress temperature relations were observed by Alers et al.[54] for molybdenum of comparable grain size. Since no twins were revealed by a metallographic examination of these specimens, it is impossible to correlate the fracture of this material with twinning, despite the fact that the fracture stresses lie approximately on a temperature plateau, which is commonly considered to be a property of twinning. At 78 K, the yield stress in compression is about 30 per cent higher than the tensile fracture stress, and a few isolated inter- and transcrystalline cracks were observed in the compression bars.
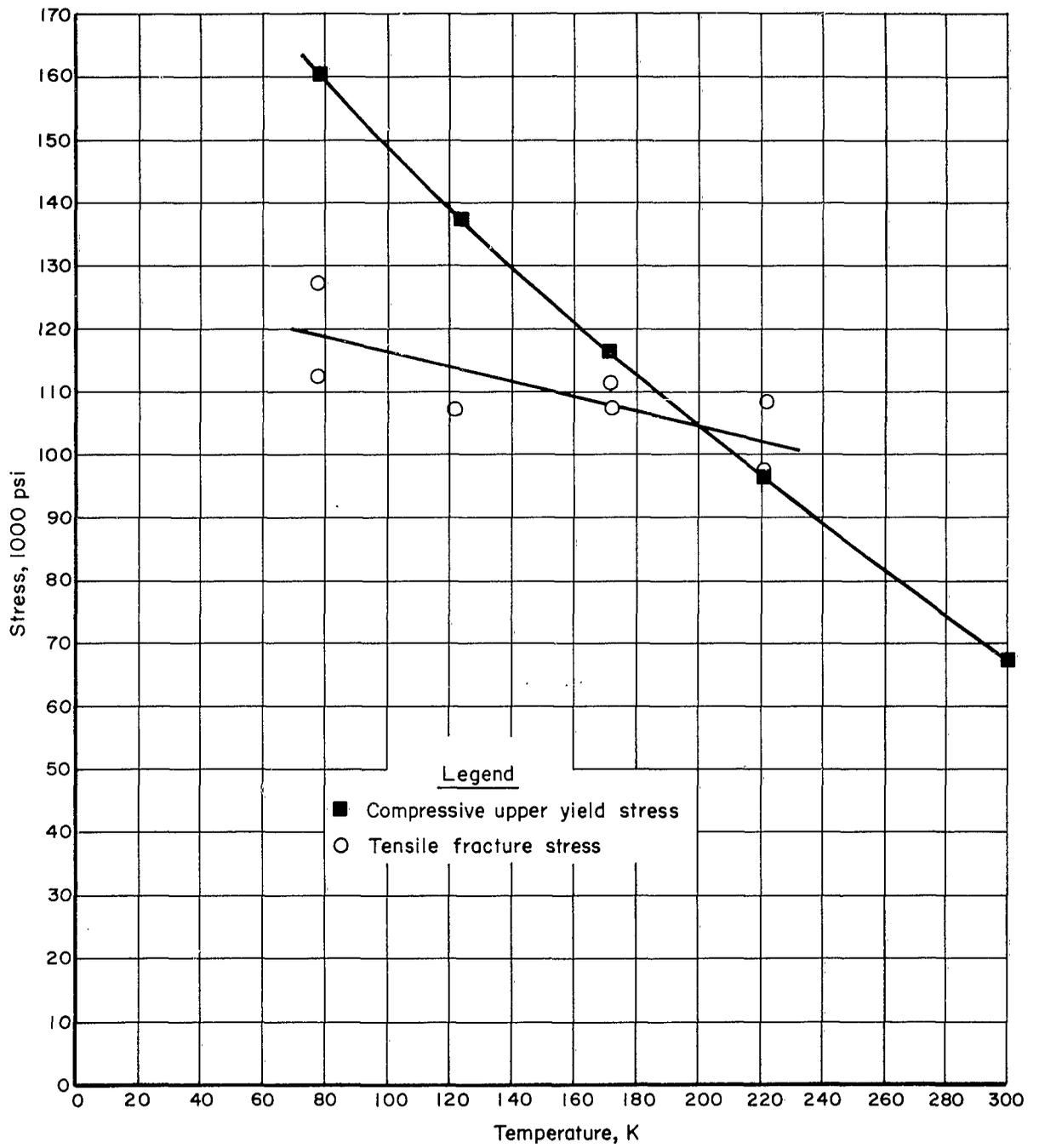
FIGURE 5. THE TEMPERATURE DEPENDENCE OF THE TENSILE FRACTURE
STRESS AND THE COMPRESSIVE YIELD STRESS FOR
MOLYBDENUM OF MEAN GRAIN DIAMETER 0.037 MM

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Tests on Molybdenum of Mean Grain Diameter d = 0.25 Mm. The behavior of molybdenum of an intermediate grain size is illustrated in Figure 6. Again, no twins were observed in any of these specimens. There is a close similarity between the fracture stress and the compressive yield stress, and this, together with the absence of twins, implies that the failures were slip induced. Examination of the fracture surface of a specimen tested at 78 K revealed both cleavage and grain-boundary facets; the failure appeared to originate at a facet of the latter kind. Specimens that were compressed below 130 K exhibited serrated stress-strain curves, and subsequently were seen to contain both inter- and transcrystalline cracks.

Since no twins were observed in the above specimens by optical microscopy, an examination was made in the electron microscope to determine if small twins were present beyond optical resolution; such twins have been cited by Sleeswyk[46] to be responsible for brittle fracture. Compression and tension specimens of the above two grain sizes were prepared for transmission electron microscopy, after deformation at 78 K. In the preparation, disks 0.03 inch thick were sliced from the specimen by grinding; these were then indented by an electrolytic jet, and subsequently thinned to the point of perforation by electrolysis. The electrolyte used was 75 per cent by volume ethyl alcohol and 25 per cent by volume sulfuric acid. In these foils, dislocations and precipitates were visible, and Figure 7 is an example of a dislocation array generated at a precipitate in a tensile specimen of intermediate grain size. However, no cases of microtwinning were ever observed.

Tests on Molybdenum of Mean Grain Diameter d = 0.40 Mm. In tests of the material with a large grain size, the tensile fracture stresses at 20 K and 78 K coincide with the compressive yield stress at 78 K to within 1 per cent. When a compression specimen was metallographically sectioned after undergoing 3 per cent strain at 78 K, it was seen to contain numerous inter- and transcrystalline cracks; a minority of these were associated with twins as illustrated in Figure 8. It is felt that the cracks have relieved stresses around the twins rather than vice versa. For example, microcracks were observed at both ends of a twin; it seems to be too fortuitous that a pair of cracks were nucleated by some other mechanism, in positions such that they were exactly bridged by a twin, and therefore it is considered that the twin nucleated one or both cracks.

A fractographic examination was made of tensile bars tested at 20 K and 78 K. The failures were composed largely of cleavage facets, with some grain-boundary facets visible. The fracture origins appeared to be intercrystalline surfaces, identified by their aplanar shape and the presence there of a second phase. When the original fracture surface of the 20 K test bar was barely removed by polishing, two types of thin markings were seen; one type was visible before etching and the other only after etching. It was concluded from this and from their appearance that both cracks and twins were located near to the main fracture. They were distributed randomly on the cross section of the specimen and were not especially associated with the region where the failure started. No twins or cracks were observed in sections of the specimen away from the fracture.

In Figure 9 the tensile fracture stress pertaining to 78 K is plotted against the inverse square root of the average grain diameter for the above three grain sizes. There is no grain-size dependence, whereas the twinning stress usually depends strongly on grain size; it is concluded that brittle fracture does not occur at the onset of twinning.
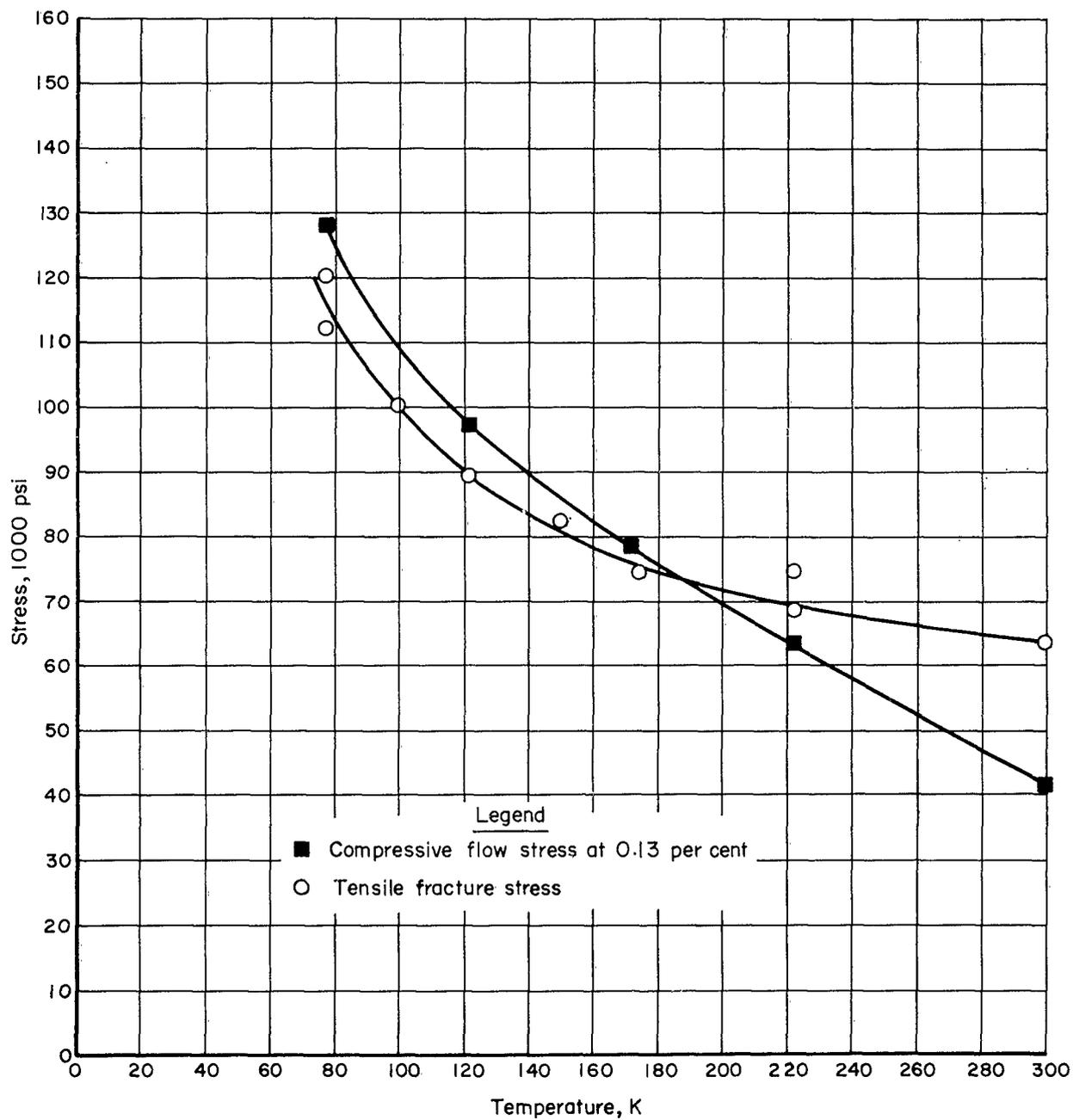
FIGURE 6. THE TEMPERATURE DEPENDENCE OF THE TENSILE FRACTURE
STRESS AND THE COMPRESSIVE FLOW STRESS FOR
MOLYBDENUM OF MEAN GRAIN DIAMETER 0.25 MM

14,000X

FIGURE 7. TRANSMISSION ELECTRON MICROGRAPH SHOWING
DISLOCATIONS GENERATED AT A PRECIPITATE
PARTICLE IN MOLYBDENUM

FIGURE 8. EXAMPLES OF CRACKS ASSOCIATED WITH TWINNING IN MOLYBDENUM OF MEAN GRAIN DIAMETER D = 0.40 MM AFTER COMPRESSION AT 78 K

500X a

500X b

500X c

500X d

17
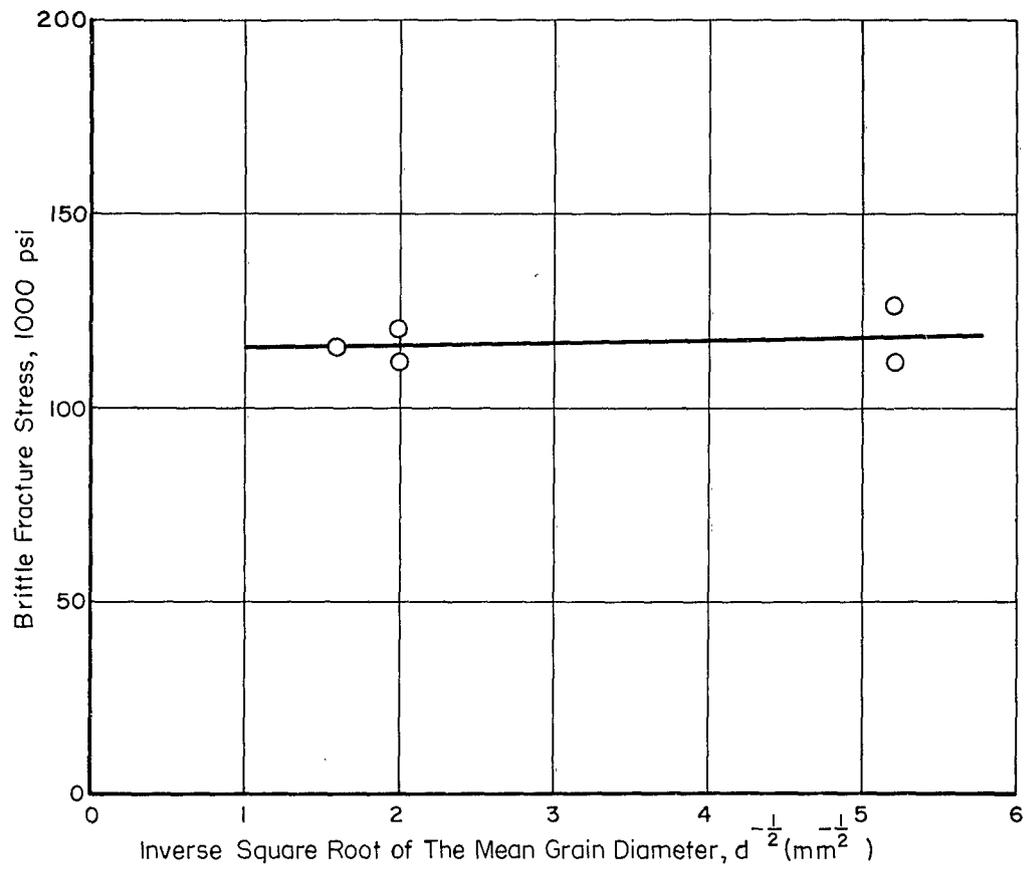
FIGURE 9. THE GRAIN-SIZE DEPENDENCE OF THE BRITTLE FRACTURE
STRESS OF MOLYBDENUM AT 78 K

Tests on Single Crystals of Molybdenum. A length of single crystal was grown from a rod of arc-cast molybdenum by the method of electron-bombardment floating-zone melting.[55] This treatment reduced the concentration of carbon from 330 ppm to 270 ppm, while oxygen and nitrogen remained at less than 10 ppm. Since little purification occurred, the molybdenum was still highly saturated with a random distribution of carbides; the principal result of this treatment was a removal of all grain boundaries and their attendant segregation.

Two compression cylinders were prepared from the crystal by a process of grinding and electropolishing. Their axial orientations are indicated in Figure 10 to be within 6 degrees of [011].

○ Columbium crystals
● Molybdenum crystals   A-44185
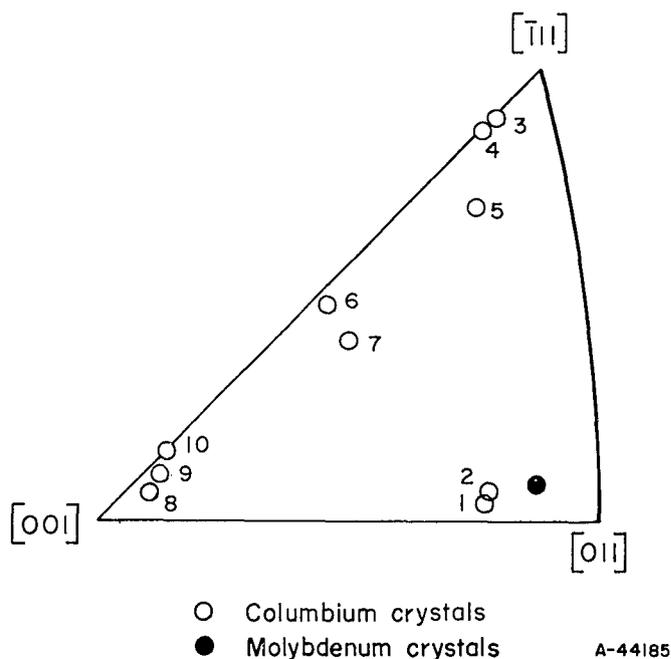
FIGURE 10. THE AXIAL ORIENTATIONS OF THE SINGLE-CRYSTAL SPECIMENS USED IN THIS INVESTIGATION

The first, Crystal A, was compressed at 78 K with a strain rate of $4 \times 10^{-2}/$ min to a strain of 1.9 per cent. Deformation proceeded entirely by slip and the stress-strain relation was smooth and monotonic with a flow stress of 108,500 psi at 0.08 per cent strain.

After being aged for 30 min at 500 C, this crystal was retested under the same conditions, whereupon profuse twinning occurred at a stress of 116,200 psi. This corresponds to a shear stress of 55,700 psi acting on the observed twinning systems. After 0.2 per cent strain, the specimen was unloaded and examined. Many cracks had developed and these were all either parallel to the specimen axis or following twin-matrix interfaces. Several interesting features were observed:

(1) Fourteen cracks were observed on a longitudinal section of this crystal and of these, as many as 11 had formed at the intersections of two twins; examples of these appear in Figure 11. The intersecting twins belong to the Schmid-Boas Twinning Systems 3 and 4. Both longitudinal and interface cracks were observed at the points of intersection.

(2) Frequently the wider twin traces contained small transverse cracks like those in Figure 11d.

(3) The center of one large longitudinal crack coincided with a subboundary for some distance; it may be seen in Figure 12a that the boundary contained some exceptionally large precipitates which may have initiated the crack.

(4) An example of the shear strain ahead of a twin lamella is seen in Figure 12b which shows the shear produced in a region of a subboundary directly ahead of a terminated twin. Sleeswyk[43] has previously observed this in iron and attributed it to emissary dislocations.

(5) Figure 12c illustrates a twin that was interrupted by a colony of precipitate particles. In three dimensions, the twin probably formed around this colony.

The second "as-grown" molybdenum crystal, Crystal B, was compressed about 2 per cent at 78 K under a strain rate of 4/min. A small number of twins formed on Systems 3 and 4, but the twinning was much less profuse than in the aged crystal. It is thought likely that the formation of these twins caused the drop in load that was observed at a tensile stress of 117,000 psi. Resolved onto Systems 3 and 4, this corresponds to a shear stress of 56,200 psi, which is in good agreement with the value for Crystal A. Intersection of twins was rare, but at one intersection of Systems 3 and 4, the small crack in Figure 12d was observed on a longitudinal section of the crystal. No other cracks were observed, either on this section or on the circumference of the specimen. It is probably significant that in these crystals the propensity of cracks is proportional to the propensity of twinning.

The difference in behavior between the two crystals is striking. It may be argued that the prestrain-age treatment received by Crystal A handicapped slip vis-a-vis twinning by first relaxing any grown-in stresses and then immobilizing all the dislocations.

In summary, the experience with the single crystals has been that 3-4 twin intersections are the major source of crack nucleation under these deformation conditions.

Results for Columbium

The mechanical properties of commercial columbium prepared by electron-beam melting have been evaluated. The material was received from the manufacturer as swaged rods with the low interstitial content specified in Table 1. Trouble was encountered inducing uniform recrystallization and eventually only one uniform grain size was produced, with a mean grain diameter of 0.38 mm. The heat treatment involved is summarized in Table 1. It has been estimated that the interstitial content that would be in solid solution after such a treatment is:[53]

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20

750X

a

750X

b

750X

c

750X

d

FIGURE 11.   EXAMPLES OF CRACKS LOCATED AT THE INTERSECTION OF TWINS ON SYSTEMS 3 AND 4 IN MOLYBDENUM SINGLE CRYSTAL A COMPRESSED AT 78 K

c. The Interruption of a Twin by a Cluster of Precipitates in Molybdenum Single Crystal A
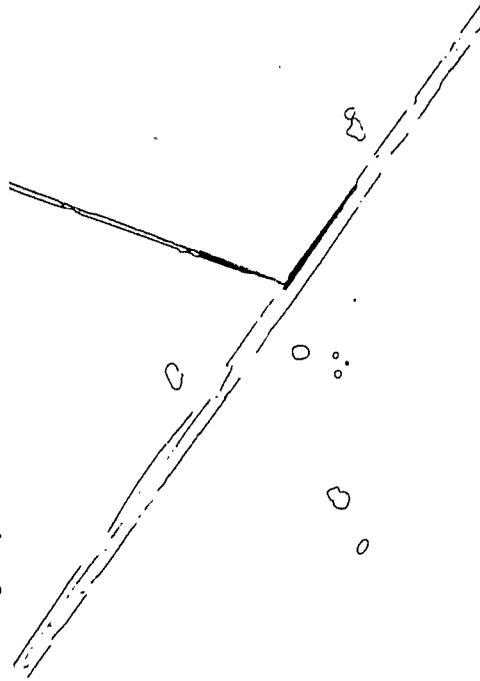
750X

d. Small Crack Situated at the Intersection of Twins on Systems 3 and 4 in Molybdenum Crystal B

750X

a. Large Crack Located at a Low-Angle Boundary in Molybdenum Crystal A

100X

b. Shear of a Low-Angle Boundary by a Nearby Terminated Twin in Molybdenum Crystal A

500X

FIGURE 12. TWINNING IN SINGLE CRYSTALS OF MOLYBDENUM

22

Hydrogen	Carbon	Nitrogen	Oxygen
9000	100	300	1000

It is concluded that all the interstitials present were in solid solution; consistent with this, no second phase was ever observed by metallographic examination.

Tests in Tension. Strained in tension at 4×10^{-2}/min, the commercial columbium was ductile at 77 K and above, deforming entirely by slip, and failing by shear with a high reduction in area (82 per cent). The stress-strain curve shows a small yield point and slow strain hardening, both of which diminish with temperature. No metallographic evidence of twinning was ever found in these tests even when the specimen was aged in situ before the test and the imposed strain rate was raised to as high as 4/min. At 20 K, however, profuse twinning occurred by deforming at a rate of 4×10^{-2}/min. After only 0.3 per cent strain, the lineal twin density (N_L) was 6.0/mm, every grain containing twins. After 7.0 per cent strain, the value of N_L was unchanged, but the average width of the traces had increased by about a factor of 1.6, implying that twin nucleation had ceased at small strains. The twins were distributed uniformly across the diameter and length of the specimens and no cracks were observed. Other investigators have observed twinning in columbium under tension only at 20 K and below.[29] After 7.0 per cent uniform deformation, a neck formed in the specimen where, at 31 per cent reduction in area, the specimen failed by cleavage. The precise point of the failure's origin could not be located by a fractographic examination, although the approximate position was determined. Within the necked region the number of twin traces was not significantly higher than in the rest of the specimen but their width was about 60 per cent greater, so it is concluded that, at the point of fracture, twin nucleation had ceased and deformation was taking place entirely by slip and growth of existing twins.

Tests in Compression. Identical material was deformed in compression at a rate of 3×10^{-2}/min. At temperatures above 120 K deformation proceeded by slip, and Figure 13 shows the excellent agreement between the upper-yield-stress (or flow stress) values obtained in tension and compression. Below 120 K, however, behavior was strikingly dissimilar; in tension, deformation occurred by slip, whereas compression caused prolific twinning throughout the entire specimen and the yield stress lay well below the value for tension. There appears to be a fundamental difference between compression and tension in promoting twinning in this polycrystalline columbium. McHargue[6] observed a similar ease of twinning in columbium under compression at 78 K, but sought to explain this solely on the basis of purity. In these experiments, however, the material is not a variable; the phenomenon occurs when two identical tension specimens are strained, one in tension, the other in compression. This negates a basic tenet of this study, viz., that it is valid and useful to correlate the behavior of polycrystalline material in tension with that in compression, and so there is a strong incentive to rationalize the phenomenon.

Slip on a given system can proceed in either sense of the slip direction, so the identical slip behavior in tension and compression is predictable. However, twinning systems are polarized, as was discussed earlier, and different systems must operate in tension and compression. In a randomly oriented polycrystalline aggregate the resolved shear stresses would favor twinning by compression in grains oriented near
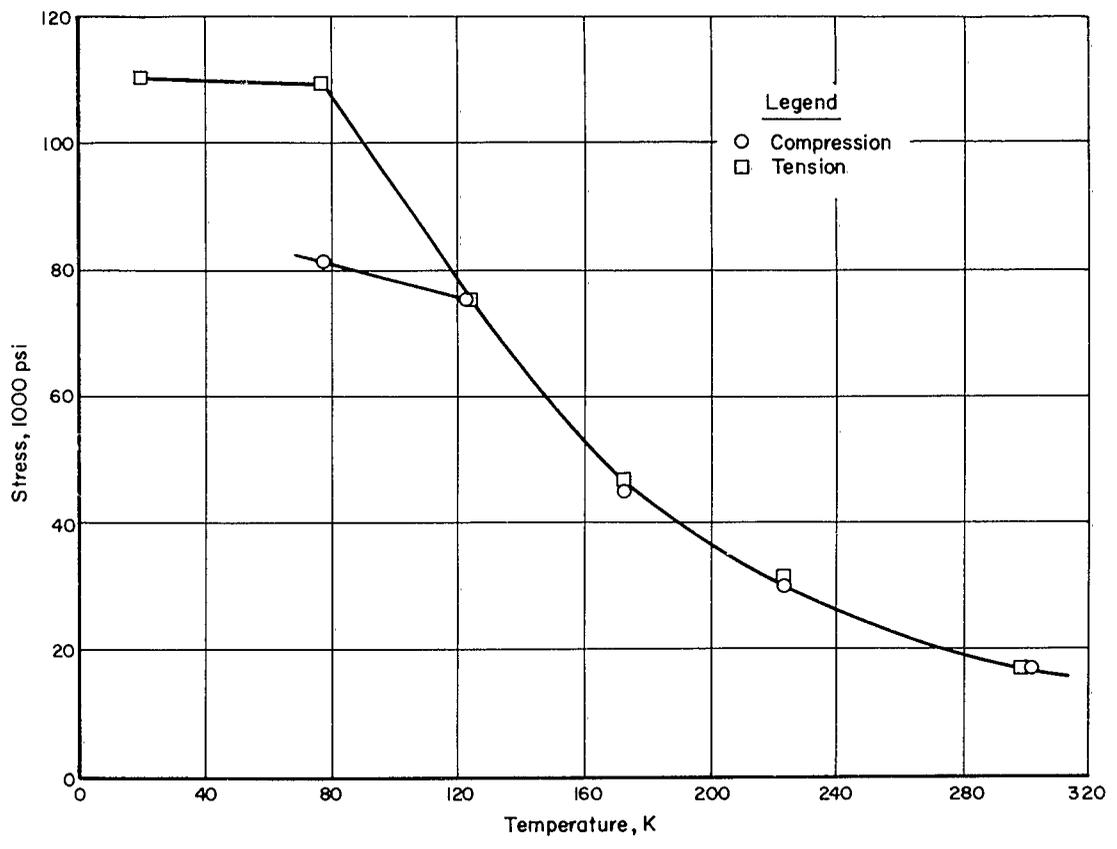
FIGURE 13. THE TEMPERATURE DEPENDENCE OF THE FLOW STRESS AT
0.2 PER CENT PLASTIC FOR COLUMBIUM TESTED IN TENSION
AND COMPRESSION

(110) and by tension in grains oriented near (100). It would be expected, therefore, that such an aggregate would show twinning in both tension and compression. That this is not observed implies that the aggregate is not randomly oriented, but has a [110] preferred orientation. This was confirmed experimentally by an X-ray method that has been described by Barrett[56]. A forward-reflection flat-plate photograph was taken of the gage of a polycrystalline tension specimen; the latter was rotated at 1 revolution/sec in order that a maximum number of grains were examined, and copper radiation was used. The measurements indicated that the specimen possessed a [110] fiber texture, combined with some cylindrical texture.[57] This texture tends to occur in a bcc metal that has undergone cold wire-drawing without intermediate heat treatment.[58] The manufacturer's report states that this material underwent the following operations:

(1) The ingot was forged at 650 C from 8- to 4-inch diameter.

(2) This bar was then rod rolled at 430 C to 1.25-inch diameter.

(3) This operation was followed by swaging at 430 C to 0.325-inch diameter.

(4) Finally, the rod was cold swaged to 0.270-inch diameter, and centerless ground to 0.250-inch diameter.

A recrystallization texture is anticipated after such intensive fabrication.

The following experiment confirmed that the texture was responsible for the observed readiness to form twins under compression. A compression cylinder was prepared with its axis transverse to the swaged rod, and its behavior was compared to that of an identical specimen machined parallel to the rod. After 6 per cent strain at 78 K the value of N_L was 16/mm in the latter sample. However, in the specimen with transverse texture a large volume of some 500 grains contained no twins whatever after this deformation. The remainder of the specimen was composed of anomalously large grains that did contain twins, but since the tendency to form twins generally increases with grain size, this can be rationalized. The important conclusion is considered to be that twinning in compression can be significantly reduced by changing the preferred orientation.

Tests of Columbium Single Crystals. The mechanical properties of columbium single crystals were studied over a wide range of orientation at 78 K. This was intended to check the above argument concerning texture that crystals with a (110) orientation tend to deform in compression by twinning and in tension by slip. Furthermore, it was desired to know the orientation dependence of twinning and of cleavage.

Tension and compression specimens were prepared by grinding and electropolishing from seeded crystals that were grown by electron-bombardment floating-zone melting. Their longitudinal axes are indicated in Figure 10. A high zoning speed of 9.7 in./hr was used in order to achieve a minimum of purification, and thereby keep the composition of the single crystals similar to that of the polycrystalline material.

Tension and compression tests were made at a rate of 4×10^{-2}/min and the stress-strain relations that were measured are reproduced in Figure 14. Crystal 1,
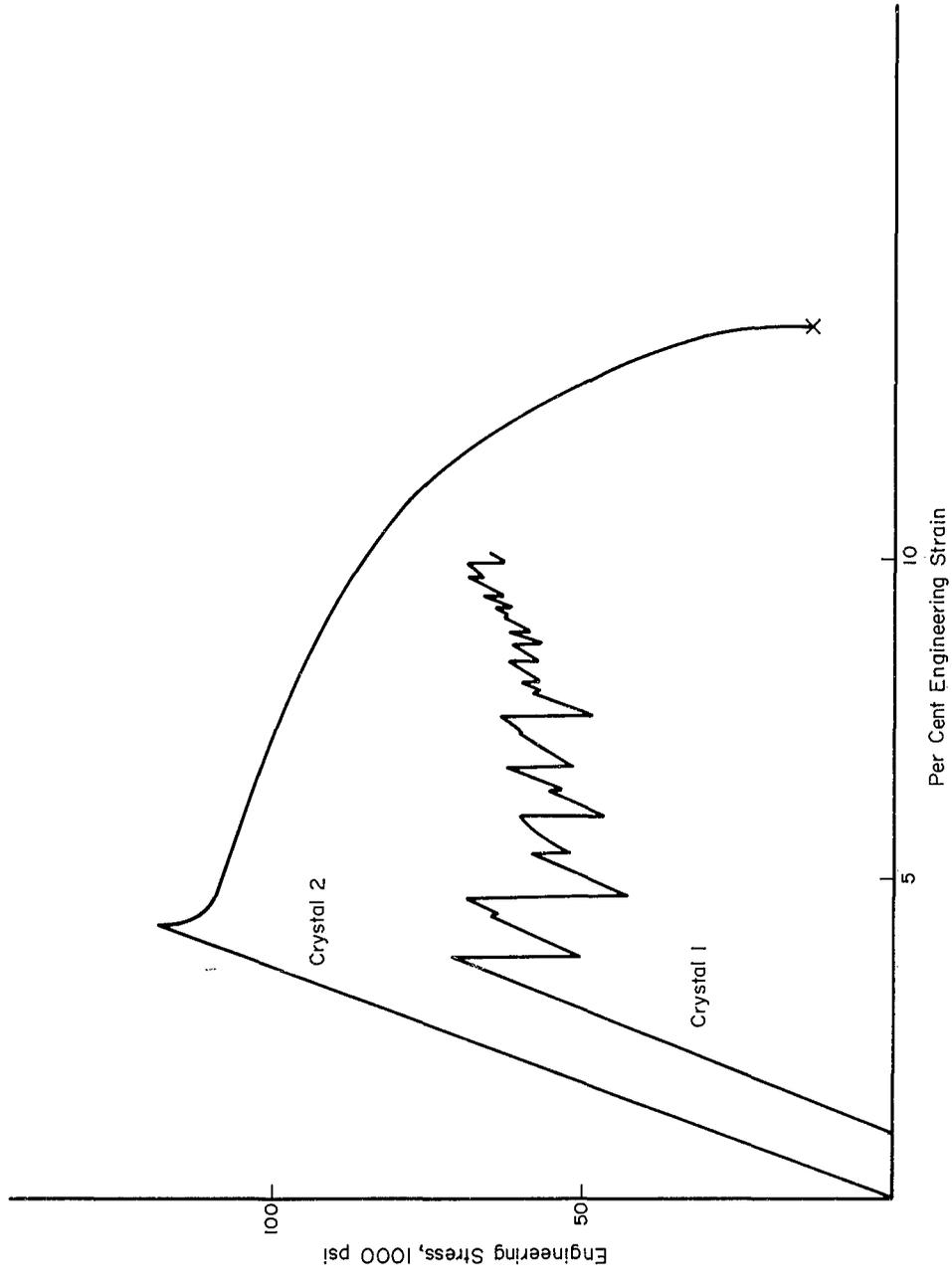
FIGURE 14. STRESS-STRAIN CURVES OF THE COLUMBIUM CRYSTALS

26

FIGURE 14. (Continued)

Per Cent Engineering Strain

Engineering Stress, 1000 psi

Crystal 3

Crystal 4

Crystal 5

FIGURE 14. (Continued)

Crystal 6

Crystal 7

Per Cent Engineering Strain

Engineering Stress, 1000 psi

Crystal 8

Crystal 10

Per Cent Engineering Strain

Engineering Stress, 1000 psi

100

50

15

10

5

FIGURE 14. (Continued)

29
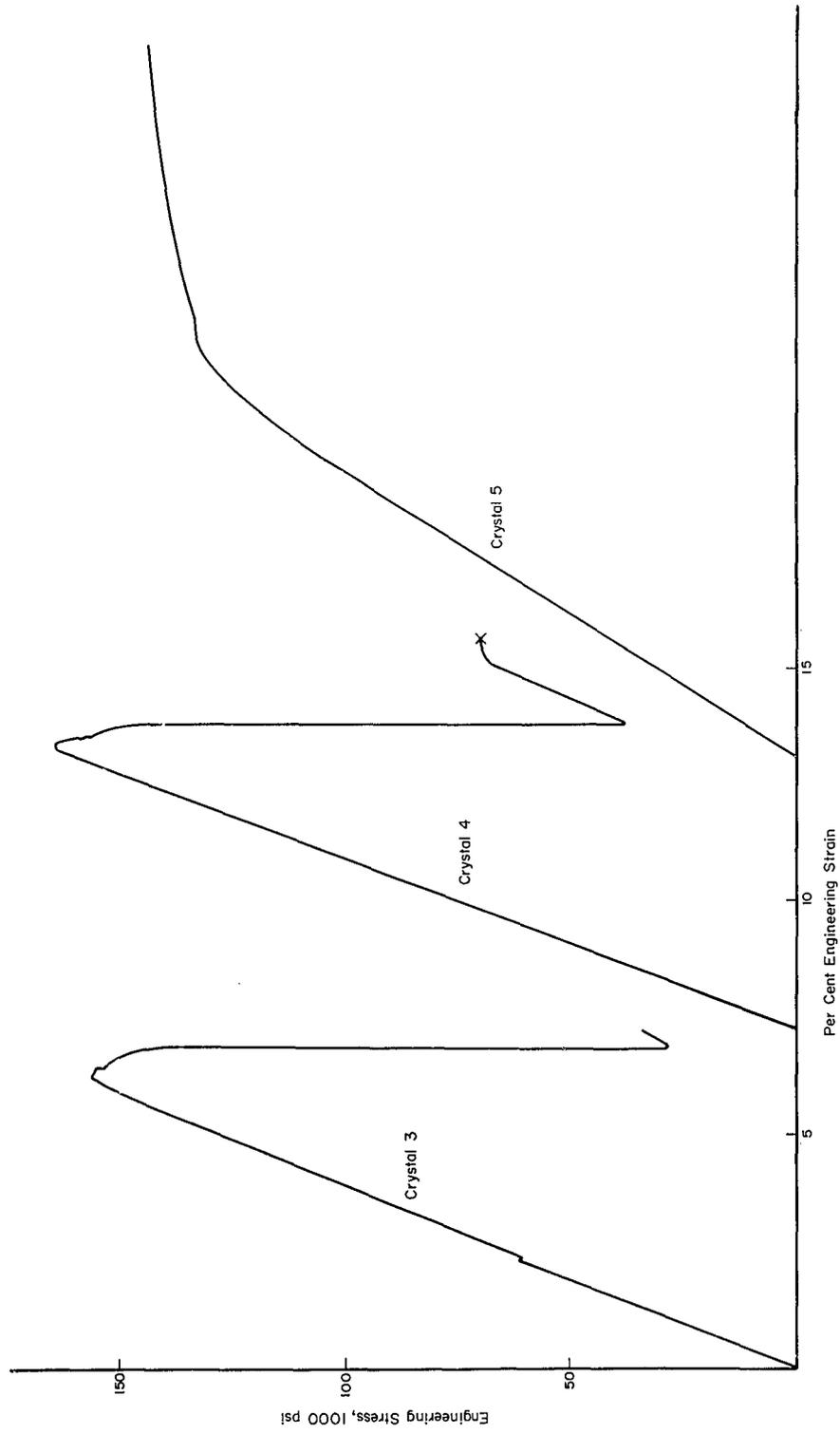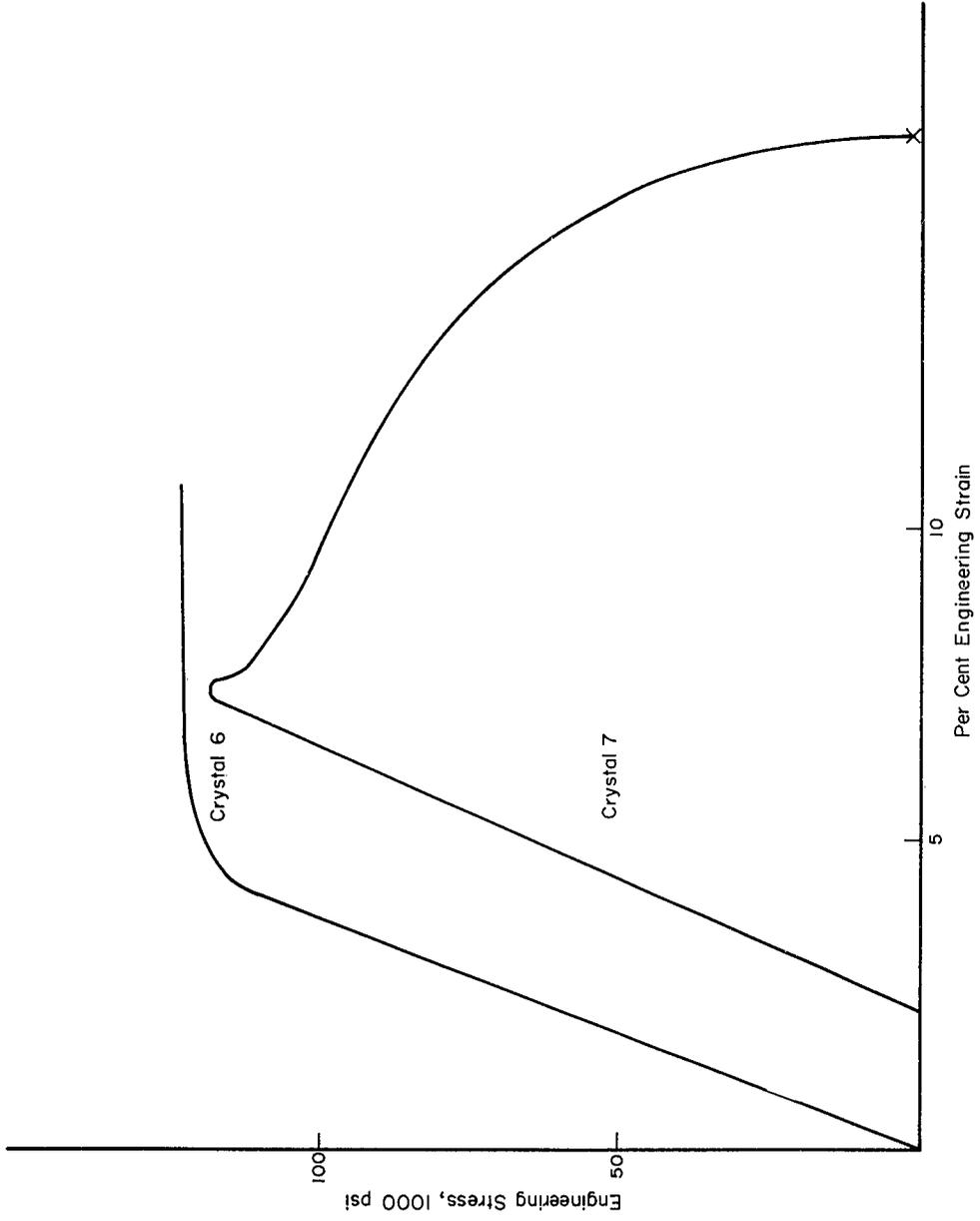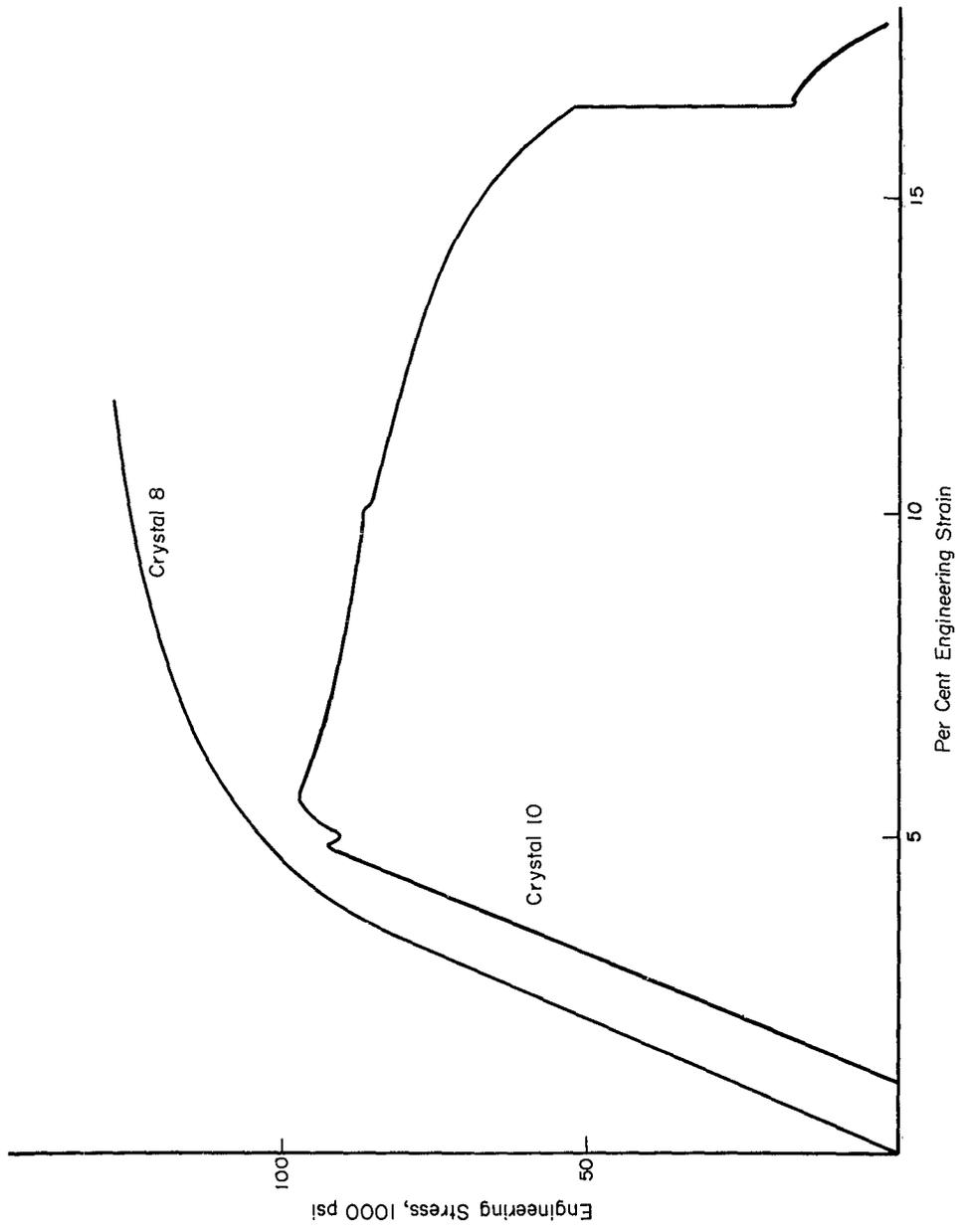
whose axis was close to [110], deformed in compression by prolific twinning on Systems 4, 3, and 6 (in order of diminishing prevalence), at a resolved shear stress of 33,800 psi. Figure 15a illustrates the extent of twinning after 6.5 per cent strain. The stress-strain curve consisted of large serrations that diminished as the strain increased. Crystal 2 of similar orientation deformed in tension entirely by slip; it displayed a yield point at a tensile stress of 113,800 psi, followed immediately by necking to a chisel edge failure. The behavior of these crystals confirms the hypothesis that a [110] texture would lead to deformation by different modes in compression and tension.

Crystals 3 and 4, oriented near [111], deformed under tension entirely by slip; no twins were observed in a metallographic examination. The slip behavior was most unusual. Crystal 3 followed the stress-strain curve illustrated in Figure 14; after a slight upper yield point, the load fell gently until at 1.5 per cent plastic strain, the load fell catastrophically by some 80 per cent. This relaxation of the specimen took place very rapidly, generating a loud noise. Following this phenomenon, the specimen behaved elastically until the test was stopped for inspection. Apparently the plastic flow had occurred predominantly in very thin layers as indicated by Figure 15b. The plane of the massive shear (although somewhat distorted) is between the plane of maximum shear stress and the nearest (110) plane, at about 12 degrees from the latter. The direction of the shear is, surprisingly, only 8 degrees from the [001] direction. It is not possible to say from this evidence whether this anomalous shear direction is due simply to slip on one unusual system, or is the vector sum of approximately equal slip in the [$\bar{1}$11] and [1$\bar{1}$1] directions. This phenomenon has the appearance of being an adiabatic plastic instability[59], except that the temperature at which it was observed is unusually high.

Crystal 4 exhibited similar behavior, as may be seen in Figure 14. The test was carried to failure, which occurred by a cleavage, associated with the massive shear step; the fracture appears in Figure 15d. The origin of the failure was traced to the central marking that is horizontal in the illustration. This marking appeared to be the line of intersection between the plane of massive shear and another slip plane whose slip lines were on the surface. Certainly it was not related to twinning, since none was observed by sectioning and etching. This unexpected case of cleavage must have been slip induced.

Upon compressing Crystal 5 of similar orientation, the flow stress was considerably smaller than the values in tension. No dynamic instabilities occurred, but the crystal showed the same preference for concentrated slip in thin lamellae, as demonstrated in Figure 15c. When slip is heterogeneous, the local strain rate, and hence the flow stress, will depend on the number of regions that are deforming. The wide range in flow stress observed between Crystals 3, 4, and 5 may be qualitatively explained in terms of observed differences in the number of regions of local slip; Crystal 5 contained the most regions, followed in descending order by Crystals 3 and 4. Consequently, the corresponding flow stresses are expected to ascent in this order; in practice the values were in the order of 127,000, 151,400, and 158,700 psi.

Crystals 6 and 7, oriented near ($\bar{1}$12), deformed entirely by slip. The flow stresses in tension and compression coincided to within 3 per cent, as expected of slip behavior. The observed rate of strain hardening was essentially zero, even under compression; this led to necking and a chisel edge failure in tension.

c. Appearance of Columbium Crystal 5 After Compression at 78 K

15X

d. Fracture Surface of Columbium Crystal 4
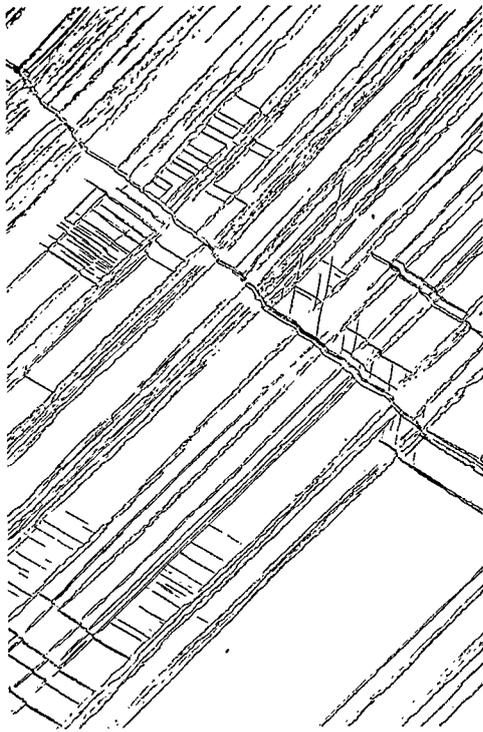
15X

a. Profuse Twinning on Systems 3, 4, and 6 in Columbium Crystal 1 After Compression at 78 K

50X

b. Shear Step in Columbium Crystal 3 After Tensile Deformation at 78 K

15X

FIGURE 15. SINGLE CRYSTALS OF COLUMBIUM

31

Similarly, Crystals 8, 9, and 10, oriented near (001), deformed entirely by slip; again, the flow stresses coincided to within 7 per cent. In the crystals tested in tension there was a tendency towards the plastic instability of Crystals 3 and 4, and several small drops in load were observed; it was established by metallographic sectioning that no twins were present. Failure in tension occurred by shear.

Experiments on the Formation and Growth of Twins

The formation of twins in specimens of Mo-35Re under stress was recorded by movie photography at speeds up to 16,000 frames/sec. Specimens of polycrystalline material, having undergone the heat treatment described in Table 1, were mounted in the grips of a small hydraulic straining device which was fitted to the stage of a metallurgical bench microscope. A carbon-arc lamp, operating from dc, illuminated the specimen, and a Fastax movie camera Model WF-1 was used to photograph the specimen surface through the microscope. Rolls of high-speed film of about 1-second duration were exposed at 6,000 and 16,000 ft/sec, during which time the specimen was strained by actuating a manual hydraulic pump. Successful records of the formation of twins were subsequently magnified further by projection. The following observations were made from these films:

(1) The first twins to form belonged to bursts; later twins sometimes formed singly. A typical burst had a value of $N_L = 5$/mm.

(2) Bursts of twins, covering many contiguous grains, became visible in the time interval of 60 μsec between adjacent frames. When first visible, the width of a twin trace was of the order of 3×10^{-4} mm, and its length generally extended across a grain, as in Figure 16a.

(3) The only growth observed was a slow increase in width of the twin traces throughout the duration of the film.

Apparently, there were two distinct phases of growth: initial and subsequent. Initial growth was too rapid to record at any intermediate stage, and therefore its mode and kinetics are unknown, except that it occurred within 60 μsec. During subsequent growth, the dimensions of the twins increased much less rapidly; meanwhile the specimen was being strained continuously, but it was not possible to correlate subsequent growth with the plastic strain, since there was no provision for measuring the latter. Other experiments were conducted in order to make this correlation.

Study of Initial Growth. Since initial growth occurs in less than 60 μsec, the phenomenon is beyond the time resolution of Battelle's movie cameras, and it was necessary to devise a faster technique. It was decided to measure the strain-time relation that accompanies twinning in a tension test, arguing that during twin growth a simultaneous increase in strain occurs, having the same kinetics. Strain was measured by an arrangement of electrical resistance gages, and their output was displayed against time on an oscilloscope; the trace was recorded by Polaroid photography. The arrangement of gages is represented in Figure 17; by using two gages bonded onto the specimen in this way, the only strains recorded were those affecting either gage alone, such as those due to localized bursts of twinning; elastic or plastic strains under both gages were ignored. The amplifier and oscilloscope were chosen as having the highest frequency response for
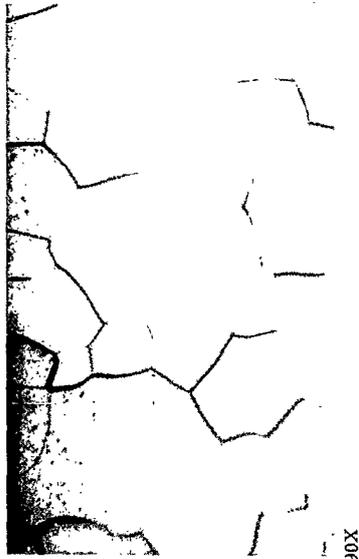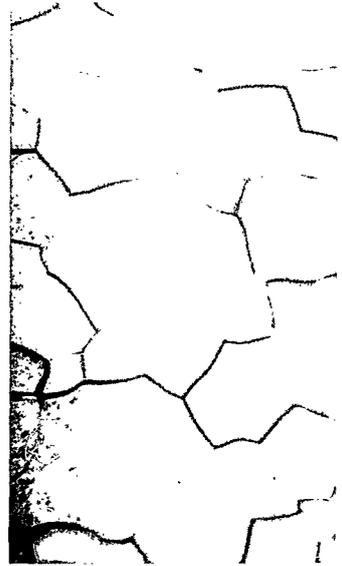
a. Adjacent Frames of the Movie Film Illustrating the Formation of a Twin Burst in Less Than 150 μsec

90X

90X
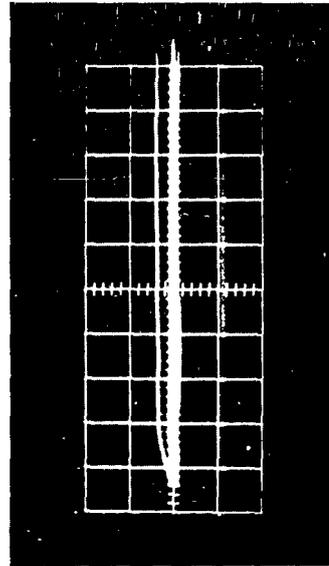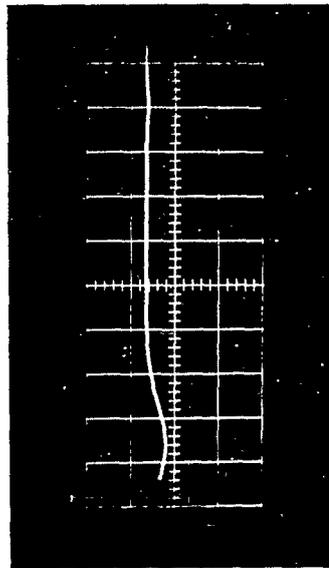
b. Two Signals From the Strain Gages Recorded on the C.R.O.

The units of the vertical scale represent about 300 μin./in., and those of the horizontal scale 1 μsec.

FIGURE 16. FORMATION OF TWINS IN Mo-35Re

a given gain, of all the apparatus available. The oscilloscope was internally triggered to display only a single sweep of the beam, so that subsequent repetitive sweeps would not fog the film.

A-40820
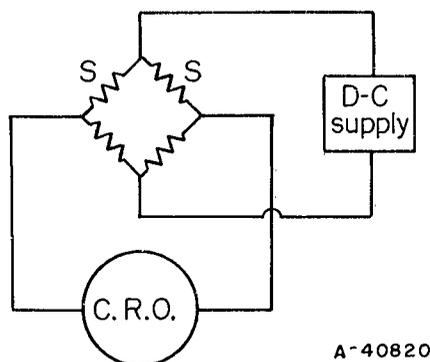
FIGURE 17. SCHEMATIC DIAGRAM OF THE CIRCUIT USING BUDD STRAIN GAGES, S, TYPE Cb-111, TO STUDY TWIN FORMATION

The C. R. O. was a Tektronix oscilloscope, Type 585, with a Type d plug-in unit; the d. c. was supplied by a Dumont strain gage controller, Type 335.

Tensile tests were made on specimens of Mo-35Re of the configuration described above. They carried two gages connected according to Figure 17, and the signals recorded are reproduced in Figure 16b. The strain-time relations consist of a step in strain, about 200 μin. /in. high, the transient of which lasts about 2 μsec. The size of this signal is consistent with that expected from a burst of twins lying partly under one gage. When step signals of known rise time were fed into the amplifier and oscilloscope it was found that the apparatus had limited frequency response; a 3 mμsec rise time was distorted to one of 1.5 μsec. Therefore, the 2 μsec rise time of the twinning signal must be regarded as a maximum figure only. It is concluded that bursts of twins form in Mo-35Re under stress in less than 2 μsec.

Study of Subsequent Growth

A study was made of the contribution of twinning to deformation as a function of the degree of strain and the strain rate used.

Tensile tests were performed at room temperature on specimens of Mo-35Re of the configuration and heat treatment summarized in Figure 4 and Table 1. Electropolished specimens were strained to about 7 per cent at a rate of 4 x 10^{-3}/min. At intervals of plastic strain, measured by fiducial marks on the specimen, the tests were interrupted and the specimens unloaded. Their surfaces were replicated by the cellulose acetate method.[60] The shadowed replicas provide a two-dimensional record of the occurrence of twinning from which the lineal density of twins, N_L, has been measured. This is presented as a function of plastic strain in Figure 18. To determine the effect of the replicating interruptions, a sample was strained continuously to about 7 per cent. The value of N_L obtained is significantly higher than that obtained in the interrupted test.

34
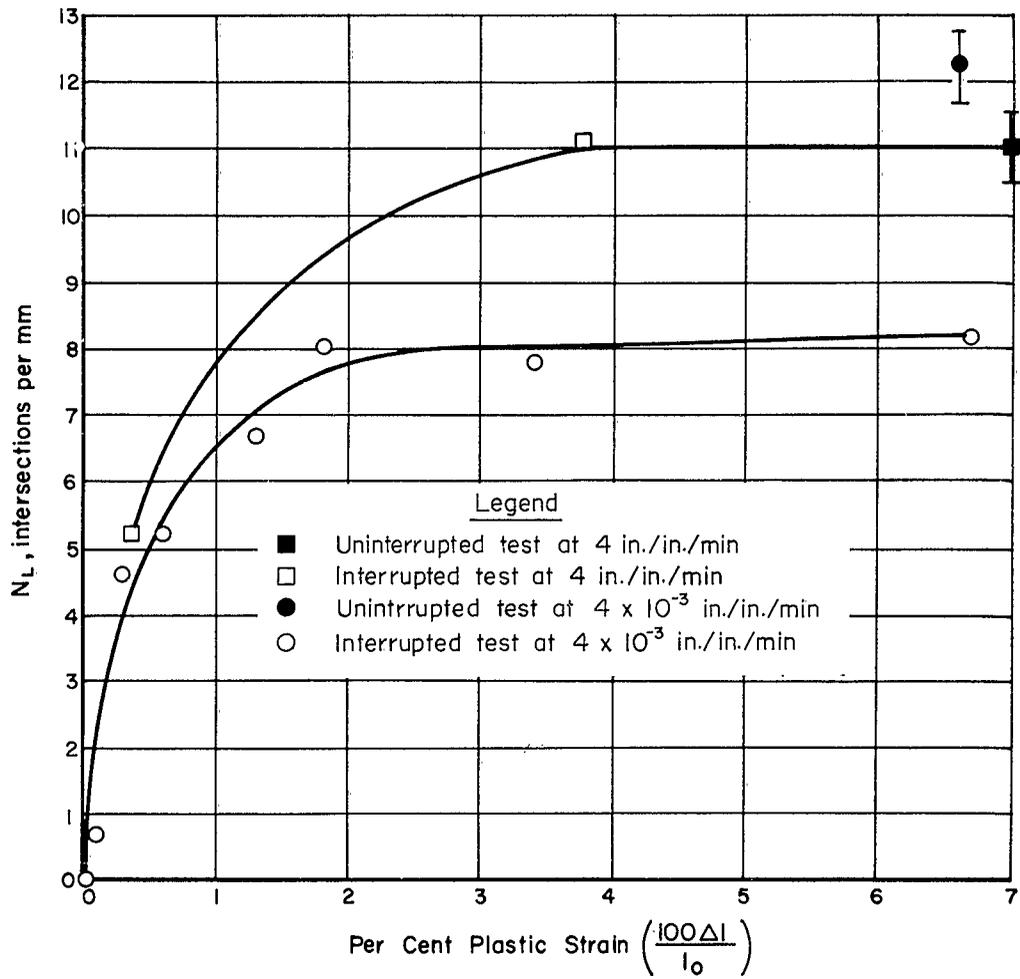
FIGURE 18. THE LINEAL DENSITY OF TWINS, N_L, IN Mo-35Re AS A FUNCTION
OF STRAIN AT ROOM TEMPERATURE

35

The effect of strain rate was subsequently evaluated. Identical specimens were strained without interruption to about 7 per cent at strain rates of 4×10^{-3} in./in./min and 4 in./in./min. The values of N_L measured on these samples agree to within the statistical uncertainty ($\pm 2X$ standard error) and appear in Figure 18. The number of twins generated is insensitive to strain rates in this range.

During straining of these specimens it was observed that existing twins increased in width. The widths of four twin traces are plotted against plastic strain in Figure 19.

From these graphs it is clear that the contribution of twinning to the deformation of Mo-35Re is almost complete at 2 per cent plastic strain, whereas twinning is theoretically able to contribute up to about 40 per cent strain. The widening of twins provides a small contribution up to at least 12 per cent strain. These results agree with the qualitative observations of Geil and Carwile[15] on twins in α-Fe.

It may be remarked that at small strains (>1/2 per cent), the rate of formation and widening of the twins accounts for most of the strain of the specimen, as computed by: (the volume fraction of twinned material) x 40 per cent.

One of the above specimens was successively sectioned to determine whether the twins seen on the surface as steps were representative of those below the surface, revealed by etching. This is a pertinent question regarding the interpretation of the movies of twin formation. In progressively polishing away the surface, N_L increased by about 30 per cent in the first 2×10^{-3}-inch depth, and thereafter stayed constant. On the other hand the average random width of the twin traces at both surface and center of the specimen was the same:

At the surface, 7.8×10^{-5} inch (81 observations)

At the center, 7.9×10^{-5} inch (50 observations).

The trace shapes visible on the surface were qualitatively the same below. For instance, in some cases serrated twin interfaces were visible on the surface, and the same serrations were also visible when the surface was polished away. It appears then that the surface tilts are reasonably representative of the interior. The slightly low value of N_L at the surface may mean that twins whose shear lies in the surface are not visible as steps, and etching is necessary to reveal them.

As a more abundant supply of Mo-35Re became available, it was decided to compare the twinning propensity of these thin sheet specimens, only 4 grains in thickness, with that of truly bulk material. Accordingly, tension and compression test bars of the type in Figure 4 were prepared by grinding. These were heat treated in the same manner as the earlier material, according to Table 1; however, the resulting grain size was only about one-half the magnitude of the earlier supply of alloy. With this variable involuntarily present, it was necessary to repeat some tests on the sheet samples for comparison with the bulk material. Sheet samples were strained in tension without interruption at a rate of 4×10^{-3}/min, each to a different value of strain between 0.5 and 6.5 per cent. The values of the lineal density of twins, N_L, are plotted against strain in Figure 20: the general relationship is the same as was found earlier, but the values of N_L are higher, and reach saturation at smaller strains. The bulk tension specimen containing about 25 grains across a diameter and the compression specimen containing about 50 grains were strained at a rate of 4×10^{-3}/min to 6.5 per cent. Their values
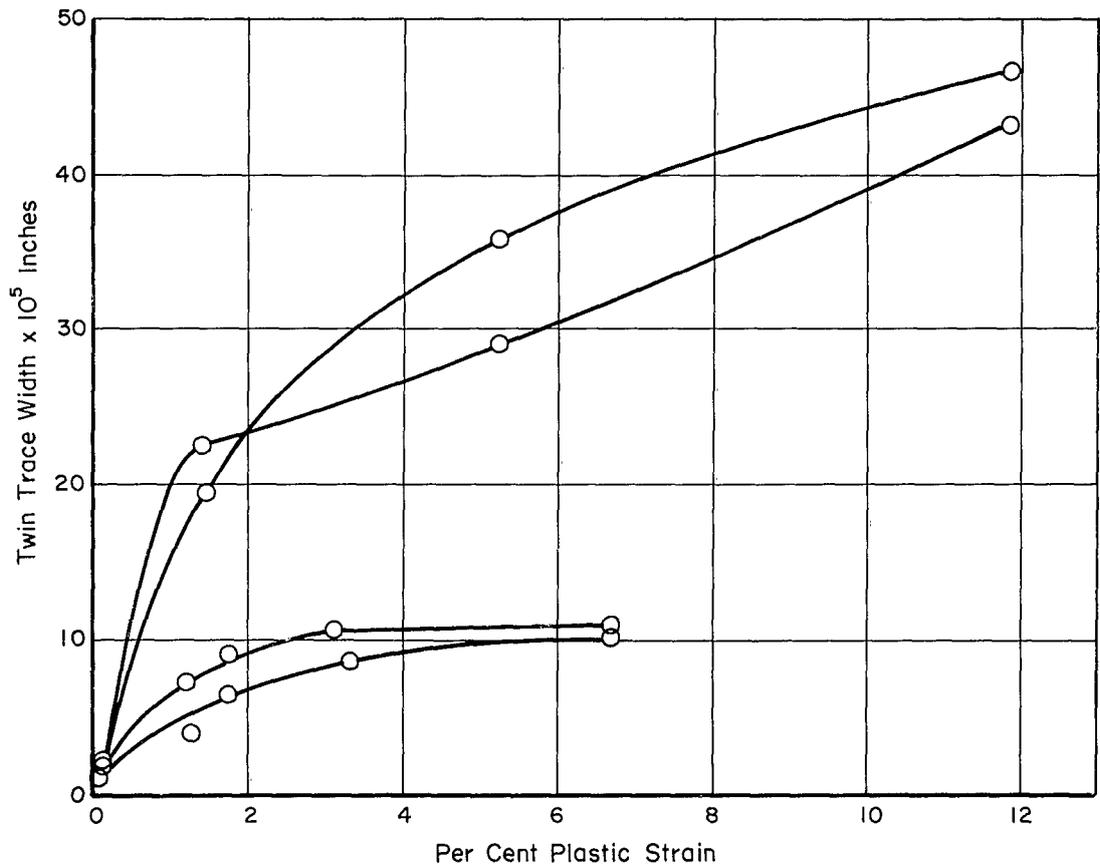
FIGURE 19. THE WIDTHS OF FOUR TYPICAL TWIN TRACES IN Mo-35Re
   AS A FUNCTION OF STRAIN AT ROOM TEMPERATURE
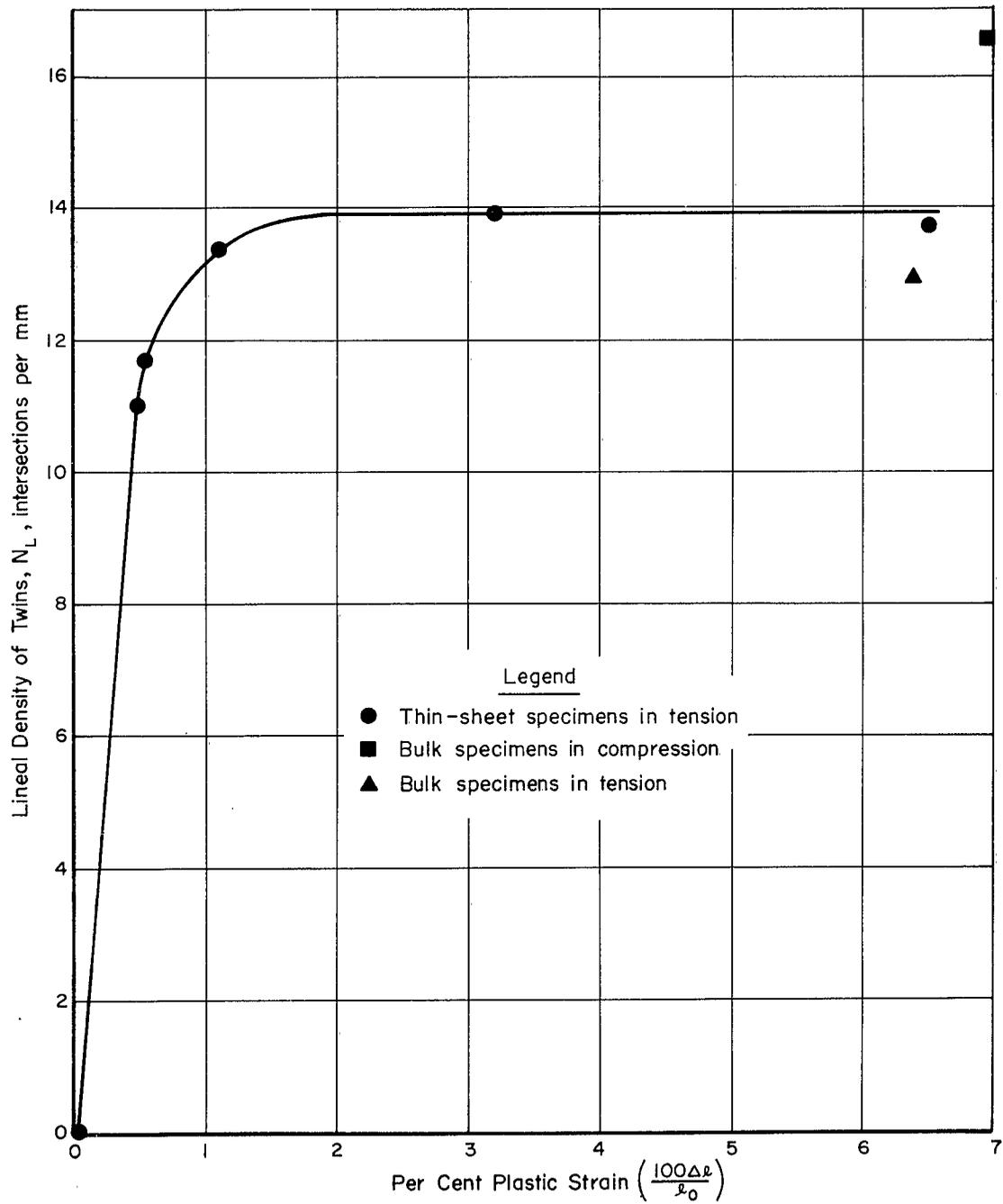
FIGURE 20. A COMPARISON OF THE EXTENT OF TWINNING IN SPECIMENS OF VARIOUS CONFIGURATIONS

of N_L are superimposed on Figure 20. There is similar twinning propensity between the thin and bulk specimens deformed in tension; bulk samples show somewhat more twinning when compressed. This may suggest the presence of some fiber texture similar to that in the polycrystalline columbium. Apparently, then, the data gained from sheet specimens is applicable also to truly three-dimensional material.

Attempts were made to observe twin nucleation by transmission electron microscopy, with a view to deciding whether it occurs homogeneously or at the sites of heterogeneities such as dislocations. Electron transparent foils of Mo-35Re were strained in tension within an RCA Model EMU 3E microscope while under observation at room temperature. Foil tensile specimens were prepared from annealed, rolled sheet of 0.010-in. thickness by erosion with an electrolytic jet to an "I"-shaped plan. The center of these foils was then thinned by electrolysis until perforation started. The electrolyte used was 75 per cent by volume ethyl alcohol and 2.5 per cent by volume sulfuric acid. Before straining, the electron transparent regions of the foils were viewed and a photographic map made. The foils were strained then manually while under observation and maps of the transparent regions were made at small increments of strain. Although twins did form in the foil they were not abundant at the magnifications used, and no case of twin formation occurred within the field of view. The dislocations observed in this alloy were unusually straight, containing few jogs or trails; frequently arrays of dislocations were generated by straining, similar to that illustrated in Figure 21.

DISCUSSION

Twin-Induced Fracture of Molybdenum

It has been clearly demonstrated above that if twins are formed in this material they are invariably coexistent with inter- or transgranular cracks. There is little doubt that the twin intersections of Figure 11 have generated the attendant cleavages, and that the impingement of twins on grain boundaries in Figure 8 has generated intergranular cracking. On this evidence, twins and their intersections are very efficient crack generators; rarely were twin intersections or twin-grain boundary junctions observed without associated cracks. This implies that the occurrence of twin-induced fracture under tension depends solely on whether the specimen in question deforms initially by twinning; it is expected that if it does, fracture will inevitably follow.

In molybdenum with a mean grain size of 0.25 mm and smaller, no twinning was observed under any conditions by optical and electron microscopy. This and the grain size-fracture stress relation of Figure 9 infer that fracture did not occur at the onset of twinning. The fracture origins identified were segments of grain boundary at which impurity segregates were visible. Apparently these sites are very effective crack nuclei, so that the material is brittle at or even below the compressive yield stress. Therefore, the mechanism seems to be that slip-induced grain-boundary rupture initiates a cleavage failure.

In the molybdenum of mean grain size 0.40 mm, twinning was observed after compression at 78 K, generating the cracks of Figure 8. However the majority of cracks observed were not associated with visible twins, and it appears that the above mechanism of crack initiation was still operative. Under tension at 20 K and 78 K this material

60,000X

FIGURE 21. TRANSMISSION ELECTRON MICROGRAPH ILLUSTRATING THE DISLOCATION
ARRANGEMENTS DEVELOPED IN ELECTRON TRANSPARENT FOILS OF Mo-35Re
BY DEFORMATION AT ROOM TEMPERATURE

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failed in a brittle manner. Again, the fracture origins were grain-boundary facets on which segregates were visible; no twinlike traces were visible on these facets, such as the conspicuous markings on the fracture surfaces of vanadium[61]. However narrow twin traces of less than $1-\mu$ width were observed across a section in the immediate vicinity of the failures. It is very difficult to decide whether these were formed prior to crack initiation or after it. It is known that twins form in very rapid bursts, and these twins may be a typical burst that preceded and perhaps triggered the failure. Alternatively since no twins were visibly associated with the fracture origin, they may merely have been generated by the stress field of the propagating failure. On the basis of available evidence it is not possible to distinguish between these alternatives. Fracture initiation appears here to be a competition between the slip and twinning mechanisms and the latter type of failure must be considered as a distinct possibility in molybdenum of this condition.

In single crystals, grain-boundary initiation of cleavage is eliminated. Thus, it is expected that the twinning mechanism of crack nucleation will be highly effective in single crystals. So far the efficacy of twins has been demonstrated only on [110] crystals under compression. However on the basis of this evidence and that reviewed earlier for iron and iron-silicon, it is expected that molybdenum crystals tested in tension along a [100] direction will also exhibit a twin-induced brittle fracture.

Twin-Induced Fracture of Columbium

There is no question of twin-induced brittle fracture of columbium at any temperature above 20 K, because all fractures were preceded by considerable plastic deformation. At temperatures above 78 K, ductile shear fractures were observed in the absence of twinning, whereas at 20 K a cleavage fracture occurred after uniform deformation of 7 per cent and 31 per cent reduction in area. In the latter case twinning contributed to the deformation, by nucleation and growth at small strains and by growth alone at high strains. Since the twins were still growing at the point of fracture, as indicated by their greater average width in the necked region, it is entirely possible that they contributed to the initiation of the cleavage failure. It is proposed to eliminate twinning by prestrain at high temperature, and observe the behavior at 20 K, to see if twins are essential to the initiation of cleavage.

The occurrence of twinning at 20 K had an interesting effect on the strain-hardening behavior. At 78 K flow became unstable as soon as yielding began, and deformation was confined to a neck. This indicates that the magnitude of the initial strain-hardening rate was less than 109,000 psi, the tensile strength. However at 20 K the formation of twins throughout the specimen induced uniform deformation up to 7 per cent strain, and a high initial strain-hardening rate of 885,000 psi was observed. Apparently the twins initiate slip uniformly throughout the specimen and postpone the instability.

The Effect of Recrystallization Texture on Twinning. It is considered that sufficient evidence has been marshalled to account for the different modes of deformation in columbium under tension and compression in terms of a recrystallization texture. First, it was established that a [110] fiber texture was present in the specimens; secondly it was demonstrated that changing the texture direction with respect to the specimen axis changes the propensity for twinning; lastly it was shown that single

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crystals with a [110] axis deform by twinning in compression and by slip in tension, which simulates the behavior of the component grains of the aggregate with a [110] texture.

This experience indicates that tension and compression behavior can be correlated only in the absence of texture. It also emphasizes the influence of preferred orientation over twinning in polycrystalline specimens. This implies that, in reporting twinning behavior and twinning stresses of polycrystalline material, it is as important to report the presence or absence of any texture as it is to report, say, the grain size. This is rarely done, complicating the direct comparison of data from different material.

The Mechanical Behavior of Columbium Crystals. From the point of view of twinning, the most significant behavior was that of the [110] crystals which deformed by twinning in compression and by slip in tension. If the columbium specimen, Crystal 1, is compared with molybdenum Crystals A and B, the difference in character between these two metals is illustrated: twinning in all these crystals was predominantly on Systems 3 and 4, but, whereas cracks were usually generated at twin intersections in molybdenum, they were never observed at the same type of intersection in columbium.

At first it appears that the behavior of Crystals 1 and 2 may be rationalized in terms of the resolved shear stress on the slip and twinning planes. In compression, the shear stress factor (cos ϕ cos λ) has a value of 0.47 for the available twinning systems, whereas in tension this value falls to 0.33. On the other hand, for slip the factor is 0.46 in both tension and compression, so the shear stress for twinning vis-a-vis that for slip is 50 per cent higher in compression than in tension. The most conspicuous inconsistency with the existence of a critical shear stress is the case of the [100] crystals deformed in tension. The values of the factor (cos λ cos ϕ) for twinning and slip systems are almost identical to those of the [110] crystal in compression. However, unlike the latter, no twinning occurred.

If the shear-stress value at which twinning started in Crystal 1 is taken as the critical shear stress, predictions can be made of the tensile stresses at which twinning would be expected to occur in the other crystals. Table 2 compares these predictions with observation. In Crystals 3, 4, and 10, slip occurred at a stress lower than predicted, and thus no check could be made. However, in all the other crystals, the predicted twinning stresses were exceeded by up to 40 per cent without any twin formation, after which deformation proceeded by slip. Clearly, the above value of a macroscopic "critical" shear stress for twinning cannot be taken seriously.

The concept of a critical shear stress for twinning was further confounded by the behavior of the molybdenum single crystals. The first crystal was compressed at 78 K up to a flow stress of 146,000 psi at 1.9 per cent strain without the formation of twins. The crystal was then aged at a temperature considered too low for dislocation rearrangement (500 C) and retested; it deformed by twinning at 117,000 psi. If this twinning stress were really critical, the crystal could not have previously sustained a much higher stress without twinning.

TABLE 2. PREDICTED TENSILE TWINNING STRESSES COMPARED WITH THE OBSERVED FLOW STRESSES

Crystal	Tensile Twinning Stress, psi, Calculated From the Observed Twinning Stress of Crystal 1	Observed Tensile Flow Stress at 0.08% Strain, psi	Observed Deformation Mode	Observed Flow Stress Resolved Onto the Optimum (110) Slip System, psi
1		71,900	Twinning	34,200
2	106,000	113,850	Slip	52,300
3	161,000	151,400	Slip	49,200
4	155,000	158,700	Slip	53,100
5	84,500	124,100	Slip	45,900
6	84,500	108,350	Slip	47,700
7	86,700	120,700	Slip	55,500
8	118,600	94,800	Slip	42,200
9	68,300	88,500	Slip	39,300
10	68,000	90,900	Slip	40,400

However, regardless of the mechanism visualized, the concept of a critical shear stress for twinning that prevails locally at twin nuclei remains a plausible hypothesis. Possibly, it is the internal stress concentrations that vary with orientation rather than the value of the shear stress.

Similarly, Table 2 shows that there is no truly critical macroscopic shear stress for slip; when the flow stresses are resolved onto the optimum (110) planes there is considerable orientation dependence, in common with many foregoing single-crystal studies. For slip, the discovery by Gilman and Johnston[62] of a relationship between the mean dislocation velocity $\bar{v}$ and the applied stress σ has rendered obsolete the former concept. They showed that, in a tensile test, the imposed strain rate $\dot{\epsilon}$ must be matched by elastic $\dot{\epsilon}_p$ and plastic $\dot{\epsilon}_p$ contributions:

$$\dot{\epsilon} = \dot{\epsilon}_e + \dot{\epsilon}_p \; . \tag{5}$$

These may be written:

$$\dot{\epsilon} = \frac{1}{M} \frac{d\sigma}{dt} + 0.5 \, b\rho\bar{v} \, (\sigma) \quad , \tag{6}$$

where M is the elastic modulus of specimen and testing machine, ρ is the density of moving dislocations, and b is their Burger's vector. In mechanical tests of single crystals there is no control over the number of moving dislocations ρ. If this were to vary among crystals of different orientations then $\bar{v}$ (σ) would have to vary inversely to maintain the equality of Equation (6); this would lead to a variety of values for σ, the flow stress. For example, the [111] crystals of columbium exhibited a marked tendency towards very localized slip, and it is likely that the number of dislocations participating in flow in this case is different from that of, say, a [011] crystal in which slip was much more uniform. It has been estimated[63] that for columbium, $\bar{v}$ may be expressed:

$$\bar{v} \, \alpha \, \sigma^{15} \; . \tag{7}$$

Since variations of up to 30 per cent are observed in the resolved flow stress from crystal to crystal, this implies that ρ may vary by up to a factor of 50. With no control over ρ, it would be old fashioned to expect a critical resolved shear stress for slip.

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The catastrophic shearing of thin layers of the [111] crystals at 78 K is a strange phenomenon from two points of view. First, the crystallography of the shear is quite unprecedented in bcc crystals, and secondly, it is unusual to observe this type of instability at such a high temperature. Since the unstable flow did not end in fracture, it was clearly not due to permanently inadequate strain hardening, like the well-known phenomenon of "necking". Also, it was established by metallographic sectioning that the instability was not due to twinning. Since the instability was only temporary, it may have been due to adiabatic heating of the specimen in the following steps. A region of the specimen is deforming normally, but due to small values of the specific heat and thermal conductivity the heat generated during deformation causes a finite rise in temperature. If the flow stress of the material is very temperature dependent, this warming of the specimen may cause an appreciable increase in the rate of deformation. This in turn will generate heat more rapidly and, if the rates of heat dissipation and work hardening do not increase correspondingly, a cyclic chain of events is initiated leading to higher and higher deformation rates. It is stopped in a hard machine when the rapidly deforming specimen relaxes the applied load. Basinski[59] has published a quantitative treatment of this type of instability; the factors promoting the instability are as follows:

(1) A large dependence of flow stress on temperature; columbium, columbium being a bcc metal, fulfills this condition, as shown by Figure 13.

(2) The concentration of flow into a small volume; the deforming lamella was only about 0.005 inch thick in these crystals.

(3) A small value of the specific heat per unit volume; if the value for columbium is interpolated at 78 K from Reference (64), it is not particularly small, being comparable with that for iron.

(4) A small value of the thermal conductivity; the value for columbium is about a quarter the value for iron at 78 K. [64]

Although columbium does not fulfill Conditions (3) and (4) especially well, it satisfies Conditions (1) and (2) very well. In fact it is perhaps the unusually localized deformation of this material that is largely responsible for the phenomenon at such an unprecedented temperature.

The highly unusual shear direction situated near [001] is most plausibly rationalized as being the net result of approximately equal slip in the conventional [$\bar{1}$11] and [1$\bar{1}$1] directions. Alternatively, the effect could be due to slip dislocations of a [001] Burger's vector. However this is considered less likely because the shear direction is actually about 9 degrees away from [001] and such dislocations have never been observed.

The Kinetics of Twin Growth

It has been established by this work that a burst of twins forms in Mo-35Re in less than $2\,\mu$sec. The formation of a twin is expected to generate a shear-stress wave, which, in propagating may initiate further twinning, leading to a burst. If this is so, then the duration of a burst is expected to be similar to the time taken by a sound wave emanating across the area of material covered by the burst. The speed of shear-stress waves, c,

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was measured in Mo-35Re by measuring the resonant frequency of a cylindrical bar attached to an Elastomat Model 500 dynamic modulus machine. The value obtained for c was 3.1×10^5 cm/sec. The bursts observed by photography covered an area of approximately 3×3 mm, and the time taken by a shear-stress wave to cross this area is $1\,\mu$sec; this value is less than $2\,\mu$sec, so the above mechanism of burst initiation by stress waves is consistent with the observations.

Figures 18 and 20 illustrate that the nucleation of twins occurs only at small strains; apparently conditions favorable for nucleation exist only at small strains, despite the fact that the nominal stress is continuously rising. Probably, after 3 per cent strain, enough mobile dislocations have been generated to continue deformation largely by slip. However, Figure 19 shows that some thickening of twin lamellae occurs up to 12 per cent strain; this is consistent with the gradual growth observed in electron transparent zinc[21] and tin[40]. The mechanism observed was the nucleation and movement of twinning partial dislocations, which, because of a small Burger's vector, are expected to occur at modest stresses. This model may well have application to Mo-35Re in bulk.

CONCLUSIONS

(1) Twin-induced cracking is observed in [011] single crystals of molybdenum-carbon and in polycrystalline molybdenum-carbon with a grain size of 0.40 mm compressed at 78 K.

(2) Fractures of molybdenum-carbon with grain sizes of 0.25 mm and smaller are by grain-boundary-induced cleavage at temperatures above 77 K.

(3) Twin-induced fracture is not observed above 20 K in high-purity columbium with a grain size of 0.38 mm, nor it is observed at 78 K in columbium single crystals of widely varied orientation.

(4) Polycrystalline columbium containing a [011] fiber texture deforms at 78 K by different modes in tension and compression.

(5) Unstable shearing of [$\bar{1}$11]-oriented columbium crystals in the [001] direction was observed at 78 K when they were deformed in tension.

(6) Bursts of twins are formed in Mo-35Re under tension at 298 K in a period of time $<2\,\mu$sec. The contribution of twinning to the deformation of Mo-35Re decreases with increasing strain.

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